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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

General reviews of progress, reagents and methods of general application.

2031. **Proceedings of the fifteenth International Congress of Pure and Applied Chemistry (Analytical Chemistry).** Lisbon. 1956. Vol. II.—This is the second of three volumes (for Vol. I see *Anal. Abstr.*, 1959, **6**, 1175) and contains 135 communications divided into three sections as follows: Section IV, Optical methods (64 papers), Section V, Radiochemical methods (34 papers) and Section VI, Organic complexes (37 papers); 21 papers have not been published elsewhere. A list of titles and authors follows, with references to journals in which the papers have appeared. The language is English unless otherwise stated. **Spectrochemical assay of anti-friction alloys,** A. Sampedro Piñeiro and E. Asensi Álvarez-Arenas, 9–28 (in Spanish) [*Rev. Cienc. Apl.*, 1957, (56), 193]. **Determination of the branching degree in natural and artificial hydrocarbon mixtures by means of the ultrasonic velocity,** M. Baccaredda and E. Butta, 29–38. **Nephelometric determination of calcium and magnesium in serum with sodium naphthalhydroxamate,** G. Beck and W. Berli, 39–42 (in German) (*Mikrochim. Acta*, 1957, 24). **Spectrographic determination of some lanthanides,** L. de Almeida, M. I. de Barros and M. R. S. Grade, 43–61 (in Portuguese). **Application to analysis of phosphorescent papers,** A. Bernanose and M. Comte, 62–69 (in French). **Flame-photometric determination of sulphate ion,** F. Burriel-Martí, J. Ramirez-Muñoz and M. L. Rexach-M. de Lizarduy, 70–110. **Interferences of three elements in flame photometry. The Cr - Co - Mn system,** F. Burriel-Martí, J. Ramirez-Muñoz and M. C. Asuncion-Omarrementeria, 111–138. **Analysis of optical glass by the atomic auto-absorption of the spectral lines,** A. Camuñas and M. Dominguez, 139–153 (in Spanish). **Ultra-violet spectrophotometric determination of phenoxathiin,** F. Capitán and R. Parellada, 154–162. **Ultra-violet spectra of carbonyl derivatives of phenothiazine with the carbonyl group directly attached to the nucleus. Determination of the position of the substituent carbonyl group,** G. Cauquil, M. A. Casadevall, E. Casadevall and M. R. Greze, 163–169 (in French). **Recording spectrophotometric titrimeter,** R. A. Chalmers and C. A. Walley, 170–178 (*Analyst*, 1957, **82**, 329). **Temperature effects in flame photometry,** R. C. Chirnside and J. E. Still, 179–190. **Chemical determination of prednisone and prednisolone in pharmaceutical preparations,** E. Cingolani and A. Gaudiano, 191 (in Italian) (short summary only). **Spectrophotometric analysis of coumarinic derivatives,** E. Cingolani, 192 (in Italian) (short summary only). **Spectrophotometric determination of quinine salts and of procaine penicillin,** F. Cotta-Ramusino and R. Intonti, 193–197 (in Italian). **Determination of vanadium spectrophotometrically in non-aqueous solution,** D. Cozzi and G. Raspi,

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for the noble metals and especially for the colorimetry of palladium, F. Burriel-Martí, F. Pino Pérez and J. F. Duchemin, 806–817 (in Spanish). **Determination of bivalent copper with benzidine-o-sulphonic acid,** F. Buscaróns and F. Nieto, 818–822 (in French). **Relation between the molecular structure and complex formation with tetrachlorophthalic anhydride,** N. P. Buu-Hoï and P. Jacquignon, 823 (in French) (short summary only). **Analytical and biochemical applications of 6:8-dichlorobenzoyleneurea,** N. P. Buu-Hoï and N. D. Xuong, 824 (in French) (short summary only). **General reaction for alkaloids,** R. Cernâtescu, M. Poni and N. Yorga, 825–829 (in French). **Complexes of indium with organic acids,** D. Cozzi and G. Raspi, 830–833 (in Italian). **Chelating agents for the extraction of lanthanide and actinide elements,** D. Dyrssen, 834–839. **Absorptiometric determination of microgram quantities of uranium using the "thoronol" complex of quadrivalent uranium,** J. K. Foreman, C. J. Riley and T. D. Smith, 840–852 (*Analyst*, 1957, **82**, 89). **Complexometric determination of aluminium,** R. Gauguin, ·853–862 (in French). **Colour reaction of ergosterol and 7-dehydrocholesterol,** A. J. A. de Gouveia, A. P. Gouveia and M. H. R. Fonseca, 863–883 (in Portuguese). **Analytical applications of organic complexes containing the pentacyanoferrate group,** E. F. G. Herington, 884–888. **Oxine and its derivatives. The sensitivity and selectivity of some 7-substituted 8-hydroxyquinoline-5-sulphonic acids and some 2-substituted 8-hydroxyquinoline-4-carboxylic acids towards certain metals,** R. G. W. Hollingshead, 889–906 (*Anal. Chim. Acta*, 1958, **19**, 447). **Steric hindrance in analytical chemistry,** H. Irving and D. Mellor, 907 (short summary only). **Structure of dithizone and its metal complexes,** H. Irving, R. P. Bell and J. Ferguson, 908 (short summary only). **Polarimetry of aspartic complexes,** K. Jacobsohn, 909–916 (in French). **Oxonol dyes as reagents for silver,** J. J. Jennen, 917–921. **Studies on the stability of some metal chelates of tiron,** R. Näsänen and J. Veivo, 922–924. **Photometric titanium reactions with polyhydric phenols,** A. Okáč and L. Sommer, 925–935 (in German). **Characterisation of the carbonyl group through complexes with mercuric chloride and cadmium chloride,** L. Paoloni and G. B. Marini-Bettolo, 936–939. **Metallic complexes and chelates of 2:4-dihydroxyacetophenone,** J. C. Pariaud, 940–947 (in French). **Copper complexes of phenyl-2-pyridylmethanol,** J. C. Pariaud and C. Tissier, 948–958 (in French). **Chelates of picolinic acid,** R. Paris and G. Thomas, 959–966 (in French). **Spectrophotometric study of the system iron(III) - lactic acid,** A. A. S. Pimenta and M. M. Silva Carmo, 967–975 (in Portuguese). **The latest researches in complexometry,** R. Přibil, 976–979 (in German). **Chelometric titrations with potentiometric end-point detection,** C. N. Reilley, 980–1010. **Metal - hydrogen peroxide - EDTA complexes and their use in analytical chemistry,** A. Ringbom, 1011–1018. **Use of a derivative of a tri-substituted di-acid as analytical reagent for sodium ions in the presence of potassium ions,** F. Salmon-Legagneur, 1019–1022 (in French). **Study on the oxalate - magnesium complex,** S. Sargin, 1023–1030 (in French). **Indirect determination of uranium by a complexometric method,** A. Sousa, 1031 (in French) (short summary only). **Metalorganic complexes of analytical significance. I. Stabilities of complexes of bivalent cations with anthranilic acid and its derivatives,** J. K. Thompson and C. L. Wilson, 1032 (short summary only). **Molecular extinction coefficients of dithizone and**

lead dithizonate in carbon tetrachloride, O. A. Weber and V. B. Vouk, 1033–1040 (in German). **Stability of copper and nickel chelates of some phthalic acid derivatives,** M. Yasuda, K. Suzuki and K. Yamasaki, 1041–1046 (*J. Phys. Chem.*, 1956, **60**, 1649).

2032. Analysis for industry. A. M. G. Macdonald. *Ind. Chem.*, 1959, **35**, 401–403; 451–453.—Recent developments in classical analytical chemistry are reviewed with respect to primary standards, indicators, new titrants and new reagents. Improvements in the determination of Hg, Cu, Ag, Mn, Be, Bi, Sc, Li and K, and in anion analysis, are discussed. The determination of CO_2 by a new method in which the gas is absorbed in acetone or pyridine and then titrated with Na methoxide, with thymol blue as indicator, is referred to. It is suggested that this method could be applied to the determination of C in organic compounds or in steel, the determination of CO_2 in air and of O after conversion into CO_2. The CO_2 content of air was determined in 15 min. with an absolute accuracy of $\pm 0.001\%$. (46 references.)
S.C.I. ABSTR.

2033. Progress in microchemistry. *Microchem. J.*, 1959, **3** (3), 275–475.—Contributions on progress in 1958 in the following subjects are published. **Reduced-scale determination of physical constants,** J. H. Badley and F. H. Stross, 275–283. **Chemical microscopy,** J. Krc, jun., 285–304. **Fractionation procedures. I. Differential migration methods,** A. G. Mistretta, 305–314; **II. Distillation, sublimation and crystallisation,** M. T. Bush, 315–321. **Qualitative inorganic analysis,** A. A. Benedetti-Pichler, 323–331. **Quantitative inorganic analysis,** R. Maurmeyer, 333–341. **Biochemical investigations,** S. Natelson, 343–398. **Elementary quantitative organic analysis,** A. Steyermark, 399–414. **Functional-group quantitative organic analysis,** T. S. Ma, 415–432. **Qualitative organic analysis,** N. D. Cheronis, 433–458. **Organic and inorganic microsynthesis,** A. R. Ronzio, 459–465. **Equipment and tools,** H. J. Francis, jun., 467–475.

2034. Accuracy in spectrochemical analysis. A. Arrak and A. J. Mitteldorf (Belmont Smelting and Refining Works, Inc., Brooklyn, N.Y., U.S.A.). *Appl. Spectroscopy*, 1959, **13** (4), 85–91.—The topics discussed include sampling, source units, electrodes, instruments, photometry and calculations. New techniques suggested for improving accuracy include devices for reducing the effects of arc wandering, the use of devices to ensure reproducible electrode packing and for traversing mechanically a flat sample during sparking, and the use of the Stallwood jet and of an enclosed arc chamber.
K. A. PROCTOR

2035. An improved phenylhydrazine reagent. W. K. Hall and T. S. Decker (Dept. Biochem., Med. College of Georgia, Augusta, U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1746.—The preparation of a stable reagent for the identification of sugars is described. Phenylhydrazine hydrochloride (20 g) and Na acetate (20 g) are dissolved in 3% sodium bisulphite soln. at room temperature, with constant stirring. For use, an equal volume of 0.2 M sugar soln. is heated with the reagent. This reagent is stable for at least 6 months, and although crystals of uncertain composition may form within this period they do not interfere with its efficiency.
S. BAAR

2036. Benzidine and its derivatives in analytical chemistry. S. J. Lyle (Univ., Durham, England). *Talanta,* 1959, **2** (4), 293–310.—A survey, based mainly on the literature between 1946 and 1958, is made of recent analytical applications of benzidine and diphenylamine and their derivatives as precipitants in the detection and colorimetric determination of oxidants, and as indicators in titrimetric analysis. When the benzidine-type molecule is used as a precipitant for anions, the ultimate accuracy depends on achieving the best balance between the solubility of the reagent and the insolubility of the pptd. salt. Other variables discussed are (*i*) the rate of development and stability of the coloured oxidation product, including the effect of *o*- and N-methyl or other groups in the reagent molecule, (*ii*) the reversibility and sensitivity of some of the reactions, and (*iii*) the determination and selection of valid oxidation potentials in relation to the indicators used. (114 references.)   W. J. BAKER

2037. Redox properties of some alkoxyl-substituted benzidines. D. I. Rees and W. I. Stephen (Chem. Dept., Univ., Birmingham, England). *Talanta,* 1959, **2** (4), 361–366.—The transitional potentials, sensitivities towards oxidants, and behaviour as indicators in redox titrations are reported for 3-methoxy-, 3-ethoxy-, 3:3′-dimethoxy- and 3:3′-diethoxy-benzidine (**I**). These compounds, which on oxidation give cherry-red or orange-red colours, are unsatisfactory for most of the titrations for which effective indicators are already available, but they can be used (especially **I**, which gives sharper end-points) in titrations of $Cr_2O_7^{2-}$ and Ce^{4+} with Fe^{3+} at acidities >4 M H_2SO_4, when normal redox indicators are unsatisfactory.   W. J. BAKER

2038. Toluene-3:4-dithiol and its derivatives as analytical reagents: a new approach to qualitative inorganic analysis. R. E. D. Clark and R. G. Neville (Cambridgeshire Tech. Coll., Cambridge, England). *J. Chem. Educ.,* 1959, **36** (8), 390–393.—Toluene-3:4-dithiol has been used for confirmatory tests for the following elements (the necessary concn. of which for good reactions are given in parentheses)—Ag (5 p.p.m.), Hg (50), Pb (5), Bi (1), Cu (1), As (1), Sb (50), Sn (1), Fe (1), Co (1), Mn (1) and Zn (50). The more strongly acidic quinoxaline-2:3-dithiol is employed for Ni (1). The use of H_2S for Cd (50) and Cr (50), catechol for Fe (1) and Cr (50), and alizarin for Al is also indicated. Diacetyldithiol acts as a catalyst in precipitations by H_2S. The reagents and experimental procedures employed are described.
O. M. WHITTON

2039. The application of the "Bio-électronique" to chemical analysis. E. P. Steimetz (Fac. of Pharm., Nancy, France). *Chim. Anal.,* 1959, **41** (8), 321–331.—A scheme of qualitative analysis is proposed which is applicable to 320 metal salts soluble in water to the extent of at least 0·1%. The substance is classified by means of eight colorimetric tests and then finally identified by measuring the pH, redox potential and resistance of a 0·1% soln. with the Bio-electronimeter (Vincent) (*Rev. Path. Comp.,* 1954, No. 663). The scheme is recommended for the identification of pharmaceutical chemicals.   W. T. CARTER

2040. Application of siloxene as chemiluminescent indicator in permanganate titrations. L. Erdey, I. Buzás and L. Pólos (Tech. Univ., Budapest,

Hungary). *Z. anal. Chem.,* 1959, **169** (3), 187–196 (in German).—Siloxene, obtained by the action of conc. HCl on calcium silicide (*cf.* Kenny and Kurtz, *Anal. Chem.,* 1950, **22**, 693), is a suitable indicator for those redox titrations in which the reaction products or the colour of the soln. obscures the end-point. It gives a yellowish-red luminescence at the end-point in the direct titration of I^-, Fe^{2+}, Sn^{2+}, Mo^{3+}, Ti^{3+}, arsenite, oxalate and H_2O_2, and in the indirect titration of IO_3^-, Ag^+ and V^{5+} with $KMnO_4$ soln. The error is generally $< \pm 1·0\%$.   G. P. COOK

2041. Use of siloxene as chemiluminescent indicator in cerimetric and chromatometric titrations. L. Erdey, I. Buzás and L. Pólos (Inst. Gen. Chem., Tech. Univ., Budapest, Hungary). *Z. anal. Chem.,* 1959, **169** (4), 263–269 (in German).—Siloxene (**I**) (50 to 100 mg in 100 to 200 ml of soln.) is a useful indicator in the cerimetric titration of oxalate, arsenite, I^-, H_2O_2 and quinol, the end-point being shown by the appearance of a yellowish-to-red luminescence (pH <3; 1·17 V redox potential). The luminescence is visible in coloured soln. **I** may similarly be used in the determination of Fe^{3+} or quinol with $K_2Cr_2O_7$ in H_2SO_4, but it consumes a little of the oxidant and a correction must be applied.   J. P. STERN

2042. Plasmocorinth B [disodium 5-chloro-2-hydroxyphenylazo-1 : 8-dihydroxynaphthalene-3 : 6-disulphonate] as indicator for complexometric titrations. G. S. Kovács and K. E. Tárnoky (Surgical. Clinic, Med. Univ., Szeged, Hungary). *Anal. Chim. Acta,* 1959, **21** (3), 297–298 (in German).—The indicator gives a blue - violet complex with EDTA in soln. containing NaOH, and an intense blue complex in soln. containing aq. NH_3. If NaOH is added to a soln. containing Ca and Mg, on titration with EDTA the titrant reacts with the Ca and then gives a colour change with the indicator. Similarly, if aq. NH_3 is added, the titrant reacts with both the Ca and Mg before reacting with the indicator. Both end-points are suitable for use in volumetric analysis.   W. T. CARTER

2043. The use of Omega Chrome Black Blue G in chelatometric titrations. A. A. Abd El Raheem and F. A. Osman (Public Health Lab., Cairo, Egypt). *Z. anal. Chem.,* 1959, **169** (5), 328–334 (in English).—Many metals give red or violet colours in soln. of pH 10 with Omega Chrome Black Blue G (C.I. Mordant Black 38) (**I**), often with high sensitivity limits. Zinc, Cd, Mn, Pb and Ni can be titrated with 0·01 M EDTA (disodium salt), with **I** as indicator, in soln. buffered to pH 10; Zn and Cd are titrated directly, while Mn needs a little ascorbic acid to prevent the pptn. of hydrated oxide, and Pb soln. should contain a little tartaric acid. With Ni the red complex with **I** is formed irreversibly, and excess of EDTA soln. is added, the excess being titrated with standard Mg soln. Soln. containing both Ca and Mg can be titrated step-wise. The Ca is first titrated in a soln. buffered to pH 12·5 by means of diethylamine, the amine is boiled off, the pH is adjusted to 10 (or better 11·5) and the Mg is titrated. Any interference due to Fe is avoided by the addition of a little KCN.   H. M.

2044. The use of sulphamic acid as a primary standard in non-aqueous titrimetry. M. M. Caso and M. Cefola (Dept. of Chem., Fordham Univ., New York, U.S.A.). *Anal. Chim. Acta,* 1959, **21** (3),

205–214 (in English).—Lithium methoxide in benzene - methanol soln. can be standardised by titration against sulphamic acid in dimethylformamide, dimethyl sulphoxide, n-butylamine or ethylenediamine. The end-point can be detected visually, with thymol blue or azo violet as indicator, or potentiometrically with platinum and calomel electrodes. The precision ranges from 0·27% in dimethylformamide to 0·5% in ethylenediamine. The solvent may contain up to 2% of water, and under normal conditions atmospheric CO_2 is without effect.   W. T. CARTER

2045. Improved preparation of sulphatoceric acid for the preparation of standard titrimetric solutions. H. Diehl and G. F. Smith (Iowa State Coll., Ames, U.S.A.). *Talanta*, 1959, **2** (4), 382–383.—Procedures are given for preparing $Ce(OH)_4$ by pptn. from aq. ammonium nitratocerate soln. and for using the air-dried product (100 mesh) (containing 2 to 3% of H_2O) to prepare a stable soln. of sulphatoceric acid (0·05 N in Ce^{IV}) in 0·75 M H_2SO_4.
                                W. J. BAKER

2046. Titration of acids in coloured solutions. A. S. Babenko. USSR Pat. No. 117,678; 6th Feb., 1959.—The acidity of coloured soln. is determined by using nitron hydrochloride (**I**) as an extraction indicator. The titration is carried out as for a normal acid - base indicator, adding 3 to 5 drops of a saturated aq. soln. of **I** and 3 ml of $CHCl_3$ for each 10 ml of soln.; near the end-point the soln. is shaken vigorously after adding each drop of alkali soln. and allowed to separate; the appearance of a golden-yellow colour in the $CHCl_3$ layer by transmitted light indicates the end-point. Good agreement is shown between results by the present method when titrating 0·1 N HCl in soln. of Cr acetate, $NiSO_4$, $CoSO_4$ and methylene blue as compared with titrations of the same amount of 0·1 N HCl with methyl orange (max. average deviation 0·02 ml on 10·26 ml), and when titrating 0·1 N tartaric acid alone, or in wine, as compared with the phenolphthalein method (average deviation 0·03 ml on 11·37 ml) and the potentiometric method (average deviations 0·04 ml on 6·69 ml and 0·03 ml on 5·27 ml).   C. D. KOPKIN

2047. The theory of titration of bases in glacial acetic acid. J. Kenttämaa and E. Heinonen (Inst. Chem., Univ. Helsinki, Finland). *Suomen Kem., B,* 1959, **32** (9), 189–193 (in English).—It is shown that the ratio of the dissociation constant of a base to that of its salt in anhydrous acetic acid can be calculated from the slope of the titration curve. The equation for the ratio is based on the equations derived by Kolthoff and Bruckenstein (*J. Amer. Chem. Soc.*, 1957, **79**, 1).   N. E.

2.—INORGANIC ANALYSIS

General, determination of elements (arranged in the order of the Periodic Table), analysis of minerals and inorganic industrial products.

2048. New spectrophotometric method for the rapid visual analysis of metals. C. Mielenz (R. Meyer & C., Torino). *Metallurg. Ital.*, 1958, **50** (8), 332–336.—Some visual spectrophotometers are described. With a high-precision model such as the Fuess 'Spektromat' it is possible to determine Si in cast iron in the range 1 to 10% with a precision of $\simeq \pm 30\%$.   J. H. WATON

2049. Metal complexes of NNN'N'-tetrakis-(2-hydroxypropyl)ethylenediamine. D. A. Keyworth (Wyandotte Chemicals Corp., Wyandotte, Mich., U.S.A.). *Talanta*, 1959, **2** (4), 383–384.—The colours, formation constants, and first and second dissociation constants, of the complexes of Cu^{1+}, Ni^{2+}, Zn^{2+}, Cd^{2+}, Pb^{2+}, Co^{2+}, Hg^{2+} and Ag^+ with NNN'N'-tetrakis-(2-hydroxypropyl)ethylenediamine are listed. The reagent is structurally similar to EDTA, but is more selective in the formation of metal complexes. Its use in complexometry, as a masking agent or titrant, and as a colorimetric reagent, is suggested.   W. J. BAKER

2050. Perbenzoic acid as volumetric reagent. I. Determination of inorganic reducing agents. Balwant Singh, S. S. Sahota and Attar Singh (Dept. of Chem., Punjab Univ., Hoshiarpur, India). *Z. anal. Chem.*, 1959, **169** (2), 106–109 (in English).—Perbenzoic acid (0·05 M) in $CHCl_3$ is used for the titration of $K_4Fe(CN)_6$, As_2O_3, potassium antimonyl tartrate, Na_2SO_3, $SnCl_2$, hydrazine sulphate and $Na_2S_2O_3$ in Na acetate - acetic acid buffer of pH 4·0 to 4·5, with iodine soln. as catalyst or pre-oxidiser. Titration is either visual to a violet end-point or potentiometric with a bright platinum foil - S.C.E. electrode system.   J. P. STERN

2051. Mercurimetry in the quantitative microanalysis of iodides, cyanides and sulphides in solution and of hydrocyanic acid and hydrogen sulphide in gases. E. Hoffmann (Div. of Building Res., C.S.I.R.O., Melbourne, Australia). *Z. anal. Chem.*, 1959, **169** (4), 258–263 (in English).—Methods described in the literature for the colorimetric determination of iodides and cyanides with $Hg(NO_3)_2$, with diphenylcarbazone as indicator, are extended to the determination of microgram quantities of HCN, S^{2-} and H_2S. In this way, I^- (4 to 7·3), CN^- (0·2 to 0·8), HCN (0·37 to 0·83), S^{2-} (0·12 to 1) and H_2S (0·42 to 1·2 μg) are determined with accuracies better than ±5%, ±9%, ±12%, ±25% and ±10%, respectively. Acidification of soln. and volatilisation of HCN or H_2S permits cyanides and sulphides to be separated from interfering ions. *Procedure*—With a gas, the sample is shaken with a measured excess of aq. $Hg(NO_3)_2$ soln. (7·5 μg of Hg per ml), 1 ml of KH_2PO_4 - Na_2HPO_4 buffer (pH 6) and 1 drop of 1% ethanolic diphenylcarbazone soln., and the soln. is matched against a blank soln. to which standard $Hg(NO_3)_2$ soln. has been added.   J. P. STERN

2052. Paper chromatography of inorganic cations with dithizone. I. Separation of copper, cadmium, mercury, lead and bismuth by precipitation paper chromatography. Hideo Nagai (Fac. of Sci., Kumamoto Univ., Kurokami-cho, Kumamoto). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (6), 617–619.—The use of dithizone (**I**) in ethanol and acetone for paper chromatography is unsuccessful because of the low solubility of metal dithizonates in these solvents. By the use of a filter-paper treated with **I** in acetone (0·5%, 100 ml for 100 sheets), Hg^{II} (orange), Cu (dark purple), Bi (orange), Cd (red) and Pb (pink) give separate circles in this order from the centre, when developed with 0·1 N HNO_3 - acetone (10:1). (For the method of development see Nagai, *Anal. Abstr.*, 1956, **3**, 2399.)   K. SAITO

2053. Volumetric determination of copper, cadmium and mercury. N. V. Koshkin. *Nauch. Dokl. Vȳssh. Shkolȳ. Khim. i Khim. Tekhnol.*, 1959,

(1), 92–96; *Ref. Zhur., Khim.*, 1959, (20), Abstr. No. 71,219.—A method based on the reaction between dithiocarbamic acid hydrazide (**I**) and iodine is presented for the iodimetric titration of micro and macro amounts of **I** in very dil. neutral or acid soln. ($\simeq$ 4 mg of **I** per 100 ml); with more concentrated soln. the reaction product colours the soln. yellow and interferes with the end-point. The titration of **I** is carried out with $0.01\ N$ iodine in KI soln. in the presence of starch. Since the complexes of **I** with Cu, Cd and Hg are only slightly sol. in H_2O and mineral acids, a method was devised for the determination of these metals, consisting in pptn. with an excess of **I** in HCl soln. and back-titration of the unchanged **I** with iodine soln. In the determination of Cu^{2+}, Cd^{2+} and Hg^{2+}, the ions of alkali and alkaline-earth metals, Zn^{2+}, Al^{3+} and Mn^{2+} do not interfere, but Ag^+, Hg^+, MnO_4^-, CrO_4^{2-} and $Cr_2O_7^{2-}$ do. The easy purification of **I** makes it very suitable for iodine titrations.   K. R. C.

2054. Polarographic determination of bismuth, antimony, lead and tin when present together. N. A. Lektorskaya and P. N. Kovalenko. *Nauch. Dokl. Vyssh. Shkoly. Khim. i Khim. Tekhnol.*, 1959, (1), 102–104; *Ref. Zhur., Khim.*, 1959, (20), Abstr. No. 71,220.—It is shown that the strength of the diffusion current of Sn, with $ZnCl_2$ as a supporting electrolyte, increases with an increase in the HCl concn. and reaches a constant value in the range 1.75 to $3.5\ N$. The addition of NaF to a soln. of Sn suppresses the diffusion current, irrespective of the HCl concn. in the range 0.3 to $3.5\ N$. With a basal soln. of a zinc salt in the presence of HCl and NaF, the height of the diffusion wave of Bi, Sb or Pb is directly proportional to the concn. of the metal. A method is suggested for the polarographic determination of Bi, Sb, Pb and Sn in zinc.   K. R. C.

2055. Electrolytic analysis by the use of chelating agents. I. Electrolytic determination of cadmium in the presence of zinc with EDTA. Genkichi Nakagawa (Inst. Tech., Gokiso-cho, Showa-ku, Nagoya). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (6), 613–616.—Whilst Cd is deposited electrolytically at pH 3.5 to 4.0, Zn is not deposited at pH >3.0 in the presence of EDTA. Since the pH markedly decreases with the deposition of Cd, a strong buffer (*e.g.*, citrate) must be used. The molar ratio of EDTA to Zn should be >1.5 for >10 mg and >10 for $\simeq1$ mg of Zn; the temp. should be 60° to 70° and the current 8 to 15 mA per sq. cm. By the use of tracers, it is shown that when Cd (0.2 to 0.5 g) is caused to deposit within 1 hr. in the presence of 30 to 100 mg of Zn under the conditions given, the Cd contains only 0.1 mg of Zn. The rate of deposition increases with increase of pH, and the time required for the deposition of <100 mg of Cd is decreased by electrolysing a soln. at pH 3.5 to 4.0.

II. Determination of copper in the presence of bismuth with diethylenetriaminepenta-acetic acid. Genkichi Nakagawa. *Ibid.*, 1959, **80** (8), 883–886.—In a weakly acid soln. containing Na acetate and diethylenetriaminepenta-acetic acid (**I**), the $E_{\frac{1}{2}}$ values of Cu (-0.10 V *vs.* the S.C.E. at pH 2.0; -0.44 V at pH 6.0) and Bi (-0.57 V at pH 2.0; <-1.0 V at pH 5.0) differ greatly. Copper ($\simeq0.1$ g) is electro-gravimetrically determined in an acetate or citrate buffer ($0.2\ M$, 150 ml) of pH 2 to 5 containing **I** ($\simeq3$ moles of **I** to 1 of Bi) and hydrazine

hydrochloride (1 g) by controlled potential electrolysis (-0.35 V) at 70°.

III. Determination of zinc in the presence of nickel and the successive determination of cadmium and zinc by the use of EDTA. Genkichi Nakagawa. *Ibid.*, 1959, **80** (9), 1015–1018.—Zinc ($\simeq0.1$ g) is quant. deposited on the cathode (terminal voltage, 1.5 to 2.0 V) from a basal electrolyte (150 to 200 ml) containing NaOH (10 to 20 g), EDTA ($0.5\ M$, 4 to 10 ml, equiv. to more than twice the amount of Ni) and hydrazine hydrochloride (2 g) in <1 hr. at 80° in the presence of Ni ($<$ twice the amount of Zn). The Zn forms a grey compact deposit, which is suitable for weighing. Cadmium (<0.5 g) is electro-deposited on a copper-plated platinum cathode from an acetate buffer ($\simeq6\%$, 150 ml) of pH 3.5 to 4.0 containing gelatin soln. (0.1%, 10 ml) and EDTA (equiv. to approx. twice the amount of Zn) in <100 min. at 60° in the presence of Zn (<0.15 g); the Zn can be subsequently determined by electrolysis.   K. SAITO

2056. Separation and determination of nickel, chromium, cobalt, iron, titanium, tungsten, molybdenum, niobium and tantalum in a high-temperature alloy by anion exchange. D. H. Wilkins (Res. Lab., Gen. Electric Co., Schenectady, N.Y., U.S.A.). *Talanta*, 1959, **2** (4), 355–360.—A dil. soln. of the sample in HF is passed through a column (8 in. by 1 in.) of a strongly basic anion-exchange resin (200 to 400 mesh, Cl^- form), which is then eluted with 2.5% HF soln. Tungsten, Ti, Mo, Nb and Ta are retained on the column, from which they can be eluted, in succession, with $8\ M$ HCl (for Ti), 10% HF - 60% HCl (for W), 20% HF - 25% HCl (for Mo), 14% NH_4Cl - 4% HF (for Nb), and 14% NH_4Cl - 4% NH_4F (for Ta). The original percolate, which may contain Ni, Mn, Al, Cr, Co and Fe, is evaporated to dryness with conc. HCl; the residue is dissolved in $9\ M$ HCl and this soln. is passed through a shorter anion-exchange column, from which Ni and Cr are eluted with $9\ M$ HCl, Co with $4\ M$ HCl and, finally, Fe with $0.5\ M$ HCl. Because Al and Mn are eluted with Ni and Cr, it is preferable first to remove Cr (as CrO_2Cl_2) from an aliquot of the sample soln. and then pass the diluted soln. through a cation-exchange resin, from which Al can be eluted with dil. HF and then Ni plus Mn with $4\ M$ HCl. The separated elements are determined by standard methods. The elution scheme is flexible, and is also adaptable for the elements in steel. W. J. BAKER

2057. Spectrographic control of rare-metals production. I. G. Yudelevich, I. R. Shelpakova, T. I. Sosnovskaya and L. S. Bortnik (All-Union Sci. Res. Mining-Metallurgy Inst. of Non-Ferrous Metals). *Zavod. Lab.*, 1959, **25** (8), 959–961.—Spectrographic methods for determining Tl, In and Te in various materials, Pb, Cd, Cu and Zn in thallium, Na and Se in tellurium, and Fe, Te and As in selenium are briefly described.   G. S. SMITH

2058. Simplified method for the spectral analysis of inert gases for impurities. O. P. Bochkova, L. P. Razumovskaya, S. E. Frish and N. V. Chernysheva. *Optics and Spectroscopy*, 1959, **6** (6), 535–536; English translation of *Optika i Spektroskopiya*, 1959, **6** (6), 818.—In the method described, which is suitable for industrial application, the gas is drawn continuously through a capillary and its emission spectrum is excited by a high-frequency discharge. The required spectral region is isolated by an appropriate filter and the selected radiation falls on a photomultiplier connected to a microammeter. The method has been applied to the

determination of N in argon, H and Ne in helium, and of Ne and He mixtures in nitrogen. Calibration is effected by the use of tubes containing standard mixtures. In favourable cases the sensitivity of the method is 0·001%.   B. S. COOPER

2059. Method and apparatus for determining the quantity of hydrogen in metal samples. National Research Corp. Brit. Pat. 827,018; date appl. 8.4.57.—The quantities of H contained in a series of metal samples are determined by evacuating an oven chamber (of fused SiO_2) and gas-collection system to a pressure $<10^{-4}$ torr and then sealing the gas-collection system from the pump. A metal sample (e.g., titanium, zirconium or uranium, or their alloys), encased in a perforated tantalum capsule, is placed in a crucible of ThO_2-coated tantalum or molybdenum contained within the oven and the sample is heated by induction to a temp. high enough to cause only H to be evolved, but below the m.p. of the sample. The H is collected in the gas-collection system, its pressure is measured by means of a McLeod or Pirani gauge, and the system is again evacuated. The crucible is then emptied in a spent-sample chamber, and returned to the oven. The procedure is repeated until all the samples have been analysed.
J. M. JACOBS

2060. Separation of alkali metals by ion-exchange resins. I. G. Venturello, C. Gualandi and I. Mazzei (Univ., Bologna, Italy). *Ann. Chim., Roma*, 1959, **49** (1), 149–168.—Mixtures of Li^+, Na^+ and K^+ can be separated on Amberlite IR-120 resin (pre-treated with 6 N HCl, then washed with water) by elution with a mixture of phenol (25 g), methanol (50 ml) and conc. HCl (25 ml) (Magee and Headridge, *Anal. Abstr.*, 1957, **4**, 2505). The eluted salts are determined with a flame spectrophotometer. The separation of Li from Na is complete, but Na and K overlap slightly. Detailed examination of the distribution curves for the eluates shows slight interaction between the ions in the mixtures, which favours the separation, the elution of K being retarded until that of Na is complete. The method is convenient for the separation and quant. determination of $\simeq 0·02$ g of each ion.   CHEM. ABSTR.

2061. Spectrographic determination of alkali metals. N. P. Egorov and I. A. Kovalev (All-Union Correspondence Inst. of Textile and Light Ind.). *Zhur. Anal. Khim.*, 1959, **14** (4), 489–490.—The ratio of the concn. of Na and K is found from the lines Na 3302·3 Å and K 4044·1 Å, with the use of copper electrodes and the lines Cu 3290·5 Å and 4022·7 Å, by the method described earlier (Kovalev, *Anal. Abstr.*, 1956, **3**, 3794); a known amount of sodium or potassium salt is then added to the soln. and the new ratio of Na to K is determined similarly. The original concn. of Na and K may then be calculated. Third components do not interfere (Kovalev, *Zavod. Lab.*, 1956, **22**, 1336).
C. D. KOPKIN

2062. Spectrographic determination of impurities in alkali and alkaline-earth metals. M. A. Notkina and S. M. Solodovnik. *Fiz. Sb. L'vov. Univ.*, 1958, [4 (9)], 341–343; *Ref. Zhur., Khim.*, 1959, (21), Abstr. No. 74,675.—The metal is converted into carbonate and mixed with powdered charcoal or sulphur, and the mixture is placed in the bore of a carbon electrode for volatilisation in an a.c. or d.c. arc; Al, Fe, Si, Mg, Mn and Cu can be determined in lithium and Cr, Fe, Cd, Si, Cu, Pb and Zn can be

determined in strontium and barium. Test conditions are tabulated together with reproducibility of results; the probable error lies between ± 10 and $\pm 12\%$ with a triplicate exposure.   K. R. C.

2063. Rapid determination of lithium in the presence of aluminium. L. Giuffrè and F. M. Capizzi (Milan Polytech., Italy). *Ann. Chim., Roma*, 1959, **49** (9), 1414–1419.—When the ratio of Li to Al is $\ll 3·5$ the soln. may be passed directly through a column containing an anion-exchange resin in the free base state; the Li is eluted with water and determined in the eluate by titration with 0·1 N HCl. For lower ratios, the soln. is first treated with an anion-exchange resin saturated with a complexing agent for the Al, e.g., K citrate or EDTA (disodium salt), and then analysed as before. The Al may be determined colorimetrically with 8-hydroxyquinoline, but in the second case it will be retained on the column and must be removed by washing with aq. NaOH soln. (5%).
L. A. O'NEILL

2064. Lithium tetra-p-tolylboron as a reagent for the quantitative determination of sodium. V. A. Sazonova and V. N. Leonov (M. V. Lomonosov Moscow State Univ.). *Zhur. Anal. Khim.*, 1959, **14** (4), 483–484.—Lithium tetra-p-tolylboron (**I**) precipitates Na^+, K^+, Rb^+ and Cs^+ from aq. soln.; K^+, Rb^+ and Cs^+ may be pptd. first with lithium tetraphenylboron, leaving Na^+ in soln. to be pptd. with **I**. The method has been tested on NaCl soln. A 10 to 13% soln. of **I** was prepared, 2 or 3 drops of 3% NaCl soln. were added to saturate the soln. with sodium tetra-p-tolylboron, and the soln. was filtered; 3 to 4 ml of the filtrate was added to 1 ml of NaCl soln. (containing 0·0301 g to 0·0454 g of NaCl), and after 5 or 10 min. the ppt. was transferred to a filter crucible, washed with the **I** soln. (1·5 to 2 ml) ($\times$ 4 or 5), dried at room temp. in a desiccator over P_2O_5 under slightly reduced pressure, then washed free from **I** with anhyd. ether and dried in a desiccator at room temp. to const. wt. The amounts of sodium determined were 39·18, 39·30, 39·23 and 39·22 % (theoretical 39·34%). By reaction with $LiBF_4$, the ppt. may be reconverted into **I**. The method may be used in the presence of Li^+. The synthesis of **I** is described.
C. D. KOPKIN

2065. Influence of organic solvents on the chromatographic separation of lithium, sodium and potassium on sulpho-resins. V. I. Gorshkov, I. A. Kuznetsov and G. M. Panchenkov (M. V. Lomonosov Moscow State Univ.). *Zhur. Anal. Khim.*, 1959, **14** (4), 417–421.—Sodium and K can be separated on Espatit-1 by using 0·1 N aq. HCl, first Na and then K being eluted; by using 0·1 N HCl in 60% acetone, Na is eluted but not K. With mixtures of Li, Na and K on Espatit-1 and aq. 0·1 N HCl as eluent, the Li and Na zones overlap; with 60% methanol as the solvent for the HCl, first Li and then Na is eluted; K remains on the resin. With 0·28 N HCl similar effects are observed, the Li and Na zones then being just separate. The K remaining on the resin is eluted with 0·3 N HCl. On KU-2, with 0·1 N aq. HCl and a Na - K mixture, the zones are just separate, but by using 0·1 N HCl in 60% methanol the separation is facilitated. Mixtures of Li, Na and K cannot be separated on SDV-3 with 0·1 N aq. HCl, but the separation is possible with 0·1 N HCl in 60% methanol. Best results are obtained by eluting Li with 0·12 N HCl in 80% methanol, Na with 0·24 N HCl in 80% methanol, and K with 0·6 N aq. HCl.   C. D. KOPKIN

2066. Spectrographic determination of sodium and calcium in Babbitt metal. N. Ya. Tarasov, E. Ya. Zatolokin and E. A. Bozhko. *Fiz. Sb. L'vov. Univ.*, 1958, [4(9)], 434–435; *Ref. Zhur., Khim.*, 1959, (21), Abstr. No. 74,644.—The spectra are excited in a low-voltage arc discharge, with a copper electrode and a 2-amp. current and are recorded with a medium spectrograph. Calibration curves of ΔS *vs.* log C are plotted for the line-pairs Ca 3181 - Pb 3220 and Na 3302 - Pb 3220 A. Babbitt-metal samples analysed by chemical methods serve as standards. The probable errors in the determination of Na and Cu are 2·2 and 3%, respectively.　　　　　　　　　K. R. C.

2067. Quantitative determination of potassium. L. Ya. Polyak. USSR Pat. No. 117,662; 6th Feb., 1959.—Potassium is pptd. as the complex salt $K_2Bi(S_2O_3)_2 . K_2NaBi(S_2O_3)_2$, and the amount of Bi in the ppt. is determined complexometrically. The reagent is an aq. soln. of a mixture (1:2) of $Na_2S_2O_3.5H_2O$ (2 molar) and $Bi(NO_3)_3.5H_2O$ (one-third molar), containing ethanol.　C. D. KOPKIN

2068. Amperometric titration of microgram amounts of copper. A. K. Zhdanov, V. A. Khadeev and T. B. Shamakhmudova (V. I. Lenin Central Asia State Univ.). *Zavod. Lab.*, 1959, **25** (9), 1036–1039.—Titration is carried out with rotating platinum micro-electrodes with dithio-oxamide as titrant. No interference is caused by Mg, Ca, Sr, Ba, Zn, Mn, Al, Pb, Cd, SO_4^{2-}, NO_3^-, Cl^- or acetic acid.　　　　　　　G. S. SMITH

2069. Coulometric titration of bivalent copper with electrogenerated bivalent tin. J. J. Lingane (Dept. of Chem., Harvard Univ., Cambridge, Mass., U.S.A.). *Anal. Chim. Acta*, 1959, **21** (3), 227–232 (in English).—The titration is carried out in a supporting electrolyte of 4 M NaBr - 0·2 M SnCl$_4$ - 0·2 N HCl at 60°, with platinum generator electrodes. The end-point can be detected potentiometrically with a platinum electrode *vs.* a S.C.E., or ampero-metrically with platinum electrodes and an applied potential of 50 to 150 mV. The average error in titrating 1·5 to 33 mg of Cu in a volume of 75 to 90 ml is $\pm0.3\%$; Zn, Sn, Pb, Ni, Mn, Co, Al, Ag, Fe^{II} and Hg do not interfere; Fe^{III} and Au^{III} are titrated with the Cu.　　　　W. T. CARTER

2070. Determination of small amounts of copper in metals with tetraethylthiuram disulphide (dicupral). J. Michal and J. Zýka (Sci. Res. Min. Inst. and Charles' Univ., Prague, Czechoslovakia). *Zhur. Anal. Khim.*, 1959, **14** (4), 422–426.—The reaction of tetraethylthiuram disulphide (I) with Cu in soln. of various acidities is studied; in fairly conc. acids the max. colour is achieved rapidly (*e.g.*, in 10 min. for 4 to 8 N H_2SO_4, 4 N HCl or 1 to 6 N H_3PO_4), but the intensity soon falls off. In more dilute acids the max. colour is reached more slowly and is less intense, but is more stable. Synthetic mixtures of the main metal and Cu, containing 25 or 50 μg of Cu in 25 or 50 ml of soln., were prepared by adding sufficient metal salt soln. to the copper soln. to give a copper content usually of 0·01 or 0·005% based on the metal, the acidity was adjusted to the optimum value, half the vol. of water, about 6 ml of 96% ethanol and 3 ml of 0·01 M I were added, and the soln. was made up to vol. with ethanol. The extinction was measured after a known time, depending on the acidity, and controls with pure Cu and Cu-free metal with the

same acidity were carried out; 0·005% of Cu in zinc could be determined in 2 to 4 N H_2SO_4, the extinction being measured after 20 min. Copper (0·005%) in aluminium was determined in N HCl (measured after 30 min.); with less than 0·01% of Cu it is best to add EDTA (disodium salt) (II). Copper (0·05%) in mercury could be determined in N HCl, measured after 20 min., II being added to mask Hg; 0·01% of Cu in tungsten was determined at pH 10·1, measured after 45 min.; 0·2% of Cu in tin (present as Sn^{IV}) was determined in 2 N HCl (measured after 20 min.); even traces of Sn^{II} reduce the intensity of the colour, owing to reduction of I. Copper (0·017%) in manganese was determined (measured after 15 min.) in 4 N H_2SO_4; 0·1% of Cu in arsenic or antimony was determined in N HCl (after 25 min.); at lower concn. of Cu, high results are obtained. Oxalic and tartaric acids do not interfere in the determination of Cu in the metals; ascorbic acid, fluorides, $Na_2B_4O_7$ and cyanides interfere.　　　　　　　　C. D. KOPKIN

2071. Complexometric titrations (chelatometry). XLII. Some further reactions of fluorescein complexones. The determination of copper and manganese. F. Vydra, R. Přibil and J. Körbl (Anal. Lab., Acad. Sci., Prague). *Coll. Czech. Chem. Commun.*, 1959, **24** (8), 2623–2627 (in German).—The reactions of fluorescein complexone (calcein) (I) with numerous cations at different pH values were studied. At higher pH values, where the indicator alone does not fluoresce, an intense fluorescence appears in the presence of Ca^{2+}, Ba^{2+}, Sr^{2+} and Mg^{2+}. At pH < 11 the fluorescence of the indicator can be observed; this increases in the presence of Zn^{2+} and Cd^{2+}, but is discharged if Fe^{3+}, Bi^{3+}, Th^{4+}, Zr^{4+}, Al^{3+}, Tl^{3+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Pb^{2+}, Pb^{3+} or Mn^{2+} are present. The reaction of I with Cu^{2+} and Mn^{2+} was used for their volumetric determination. *Determination of Mn*—To a slightly acid soln. of Mn^{2+} (3 to 51 mg) add a small amount of hydroxylamine and triethanolamine hydrochloride, and 3 ml of pyridine or 10 ml of M ammonium acetate per 100 ml of soln.; add a few mg of indicator (1 part of the Na salt of I with 100 parts of KCl) and titrate at pH 8 with 0·05 M EDTA (disodium salt) (II) till the yellow - green fluorescence appears; Mn^{2+} can also be determined at pH 10 to 11 with the use of NH$_4$Cl - aq. NH$_3$ buffer soln. or aq. NH$_3$ (1:1) and triethanolamine; Al and Fe^{3+} do not interfere. *Determination of Cu*—From 3 to 63 mg of Cu^{2+} can be titrated at pH 10 (10 ml of NH$_4$Cl - aq. NH$_3$ buffer soln. or 5 ml of aq. NH$_3$ soln. per 100 ml) with 0·05 M II.　　　　　　　　　　J. ZÝKA

2072. Separation of copper and bismuth by electrodeposition with an amalgam - copper cathode. P. N. Kovalenko and V. L. Dmitrieva. *Nauch. Dokl. Vyssh. Shkolý, Khim. i Khim. Tekhnol.*, 1959, (1), 97–101; *Ref. Zhur., Khim.*, 1959, (20), Abstr. No. 71,215.—By increasing the pH, the recovery potentials of Cu and Bi in a nitrate - tartrate medium with an amalgam - copper cathode are shifted to negative values, but on raising the temp. they are shifted to positive values. The decomposition voltages of salts of Cu and Bi are markedly decreased by raising the soln. temp. An increase in the Na tartrate concn. produces no appreciable effect on the recovery potential or the decomposition voltage of copper salts, but with bismuth salts the decomposition voltage is raised and the recovery potential becomes more negative; this makes possible a separation by electro-deposition of Cu

and Bi. The optimum conditions for the separation of Cu and Bi are—electrolyte, 0·5 N Na tartrate, pH 4 to 5, and temp. 60°.   K. R. C.

2073. Sub-micron determination of silver iodide. L. R. Koenig (Cloud Physics Lab., Dept. Meteorology, Univ. of Chicago, Ill., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1732-1735.—When AgI is reduced in the photographic process, the resulting silver has a unique filamentary appearance. This has been made the basis of a test for AgI particles from commercial silver iodide aerosol generators and for AgI in ice crystals for cloud 'seeding', in which the particle is mounted on an electron-microscope specimen-screen and allowed to react with a photographic reducing soln. for 15 min. (2 vol. each of satd. aq. soln. of metol and Na₂SO₃, and 1 vol. each of satd. aq. soln. of quinol and Na₂CO₃). After being washed in water and dried, the specimen is examined with an electron microscope and the length of the silver filament is determined. The dimensions of the original AgI particle may be calculated from this, and the method has been applied to as little as 3×10^{-17} g of AgI. It is claimed that the method can be extended to cover AgBr and AgCl.   T. R. ANDREW

2074. Polarography of beryllium. K. Győrbíró (Dept. of Inorg. Chem., Tech. Univ., Budapest). *Magyar Kém. Foly.*, 1959, **65** (9), 354-357.—In a basal soln. 0·2 M in MgCl₂, 0·1 M to 0·2 M in tetramethylammonium iodide or (best) 0·1 M to 0·2 M in LiCl, salts of Be give two polarographic waves between pH 3·5 and 4·4. If the concn. of the Be soln. is $0·8 \times 10^{-4}$ M to 6×10^{-3} M, the height of the second wave is proportional to the concn. of Be and, in the pH range mentioned, is suitable for analytical use. According to the pH, $E_{\frac{1}{2}} = -1·85$ to $-1·95$ V; lowering the pH shifts it towards the more negative value. The position of the wave does not alter in the presence of complexing agents. Beryllium cannot be separated polarographically from Ba and Al, but Na and K do not interfere, except when present in very large excess. On the basis of the Ilkovič equation the electrode process corresponds to a two-electron reduction; presumably two hydrogen atoms of $[\text{Be}(\text{H}_2\text{O})_4]^{2+}$ are reduced.   A. G. PETO

2075. Determination of beryllium by the photo-neutron method. II. A study of methods for overcoming interferences. G. W. C. Milner, J. W. Edwards and W. M. Henry (Chem. Div., A.E.R.E., Harwell, Berks., England). A.E.R.E. Report AERE-R 3212, 1960, 19 pp.—The present report describes experiments to overcome the interference in the previously published method (Milner and Edwards, *Anal. Abstr.*, 1960, **7**, 891) from elements with high cross-sections for thermal neutrons. The approaches considered were (*i*) the use of a cadmium shield (0·1 in. thick) to prevent thermal neutrons from reaching the detectors, (*ii*) the use of a standard addition of Be to the sample, and (*iii*) reducing the thickness of the sample. The preferred method was (*i*), which resulted in the elimination of the interference in the examination of both solids and liquids. A detailed description of the use of a modified apparatus, which has resulted in only a 10% reduction in sensitivity, is given.
    G. J. HUNTER

2076. Spectrographic detection of trace impurities in beryllium oxide. O. P. Killeen (Union Carbide Nucl. Co. Y-12 Plant, Oak Ridge, Tenn.). *U.S.*

Atomic Energy Comm., Rep. Y-1259, Aug., 1959. 12 pp.—Tentative methods are described in this interim report. The elements detected include the rare-earth elements Er, Tm, Eu, Dy, Gd and Sm, and the common elements Co, Au, Fe, B, Si, Mn, Pb, Cr, Al, Ca, Y, Pd, Cu, In, Cd, Ag, Ti, Rh, Ni and Li. The observed detection limit for each element is reported.   NUCL. SCI. ABSTR.

2077. Rapid photometric determination of low-level magnesium in rocks. L. Shapiro (U.S. Geological Survey, Washington, D.C.). *Chemist Analyst*, 1959, **48** (3), 73-74.—The method described is suitable for the determination of Mg in rocks containing from 0·01 to 2% of MgO and up to several hundred times as much CaO. Phosphate rocks can be analysed after separation of either the Ca or the phosphate. Thiazole yellow (C.I. Direct Yellow 9) is used as the colour-forming reagent and interfering elements are complexed with aq. cyanide - triethanolamine soln.   G. S. ROBERTS

2078. Studies in polarographic analysis. XXXIII. Determination of calcium and magnesium. Masayoshi Ishibashi, Taitiro Fujinaga and Toyoshi Nagai (Chem. Dept., Fac. Sci., Kyoto Univ., Sakyo-ku). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (5), 482-485.—The polarographic method for the determination of Ca in serum in the presence of the Zn - EDTA complex (Přibil and Roubal, *Coll. Czech. Chem. Commun.*, 1954, **19**, 252) was modified so as to cause both Mg and Ca to react with Zn - EDTA in an ammoniacal soln. containing NH₄Cl (4·5%) and thus the sum of the Ca and Mg could be found. By combination with the original method, both elements are determined with an error of < 3%. Polyacrylamide (used as a max. suppressor) (Kolthoff *et al.*, *Anal. Chim. Acta*, 1958, **18**, 295) interacts with Zn. The sample soln. is made up to 10 ml with Zn - EDTA ($6·7 \times 10^{-3}$ M, containing a slight excess of Zn, in 8 N aq. NH₄), satd. NH₄Cl soln. (1 ml) and gelatin soln. (0·2%, 1 ml) and the polarogram is recorded.   K. SAITO

2079. Investigation of the possibility of the determination of strontium by flame spectrophotometry. J. Debras and I. A. Voinovich. *Bull. Soc. Franç. Céram.*, 1959, (42), 43.—The determination of Sr in materials in which Ca, Mg and possibly Ba are also present has been investigated by means of flame spectrophotometry. Experiments show that pure Sr in soln. can be determined by this technique, over a range from 2 to 100 mg of Sr per litre, by using the spectral line 4607·3 A. The determination is affected by the presence of Ca; a large excess of Ca, however, has a "buffer effect" and in some cases allows the required analysis to be completed. The emission of Sr is almost completely inhibited by the presence of Al; Fe, Ti, Na, K, Li and Ba affect it in various ways. The emission sensitivity of Sr is increased by the presence of EDTA and is also protected by it, although imperfectly, against the effect of the interfering elements. A considerable "anion effect" is exerted by SO₄²⁻, PO₄³⁻, Cl⁻, NO₃⁻ and OH⁻, contrary to what has been observed with Na, K and Li.   BRIT. CERAM. SOC. ABSTR.

2080. Separation of strontium from barium ions by paper electrochromatography. Kuan Pan, Hwasheng Cheng and Yuan-Tzer Lee (Dept. of Chem., Nat. Taiwan Univ., China). *J. Chinese Chem. Soc.*, Ser. II, 1959, **6** (1), 47-54 (in English).

—The complete separation of Ba^{2+} and Sr^{2+} is possible by paper electrochromatography of soln. of the ions in aq. citric acid soln. containing methanol, ethanol or *isopropyl* alcohol. The relative velocity of the ions increases with increasing concn. of alcohol, the Sr^{2+} migrating ahead of the Ba^{2+}.

G. S. ROBERTS

2081. **Determination of barium as sulphate.** J. Agterdenbos (Lab. for Anal. Chem., Univ. of Amsterdam, Netherlands). *Z. anal. Chem.*, 1959, **169** (2), 81–88 (in German).—Addition of ethanol or acetone (100 ml) to the soln. of barium (100 mg) in $2 N$ HCl (100 to 200 ml) before normal pptn. with $2 N$ H_2SO_4 results in quant. pptn. of $BaSO_4$ within 15 min. The usual method (from hot dil. soln.) applied in the presence of Fe^{3+} yields a ppt. containing up to 0·6% of Fe; the ppt. obtained from cold conc. soln. or by the use of $K_2S_2O_8$ as a scource of SO_4^{2-} contains less Fe^{3+} (0·2%).

J. P. STERN

2082. **Precipitation of barium chromate from homogeneous solution using complexation and replacement. Separation of barium from relatively large amounts of strontium and lead.** F. H. Firsching (Univ. of Georgia, Athens, U.S.A.). *Talanta*, 1959, **2** (4), 326–331.—The Ba is pptd. by K_2CrO_4 at pH 8 to 10 and 90° to 95°, from a soln. containing an excess of EDTA (disodium salt) (sufficient to complex all the Ba and all the cations present that form more stable EDTA complexes than Ba), by the dropwise addition of an excess of Mg^{2+} (as $MgCl_2$) to the soln. over a period of $\simeq 1$ hr. The Mg^{2+} gradually replace Ba^{2+} in the EDTA complex, so that cryst. $BaCrO_4$ is slowly and uniformly pptd. with a minimum of co-pptn. By the use of radioactive tracers it is shown that, when Ba and Sr are present in equi-molar concn., $>99\cdot7\%$ of Ba with $<0\cdot6\%$ of Sr can be pptd. There is no co-pptn. of Pb at any time. The sample soln. should preferably contain $\simeq 70$ mg of Ba; the EDTA must be present in excess before the addition of a twofold excess of K_2CrO_4, and enough Mg^{2+} must be used to combine with all the excess of EDTA and also to replace all the Ba in the complex. This procedure will also ensure complete separation of Ba from Ca and Fe.

W. J. BAKER

2083. **Extraction of zinc with tri(*isooctyl*)amine in *isobutyl* methyl ketone, and colorimetric determination with zincon in the organic phase.** L. E. Scroggie and J. A. Dean (Dept. of Chem., Univ. of Tennessee, Knoxville, U.S.A.) *Anal. Chim. Acta*, 1959, **21** (3), 282–288.—The extraction procedure of Mahlman *et al.* (*cf. Anal. Abstr.*, 1955, **2**, 1225) is utilised for the rapid determination of traces of Zn in alloys. *Procedure*—To an aliquot of the sample soln. containing 30 to 150 μg of Zn add $10 N$ HCl (1 ml) and dilute to 5 ml. Extract with tri(*isooctyl*)amine in *isobutyl* methyl ketone (5%, w/v) (10 ml) for 3 min. Transfer the ketone layer to a 50-ml flask, add N KOH (5·0 ml), *iso*butyl methyl ketone (5·0 ml) and $0\cdot002 M$ zincon (3·0 ml). Dilute to volume with ethanol, mix, and set aside for 8 min. Measure the extinction at 620 mμ against a reagent blank and read off the Zn concn. from a previously prepared graph. Several heavy metals, including Cu, Co^{II}, Cr, Cd, Fe, Ni, Sn and V, interfere. Six analyses of a Mg-based alloy containing 1·03% of Zn gave a standard deviation of 0·03, and six analyses of an Al-based alloy containing 0·076% of Zn gave a standard deviation of 0·003.

W. T. CARTER

2084. **Spectrographic control of zinc-plating baths for steel strips by the Sendzimir process.** A. Porta (Controllo Qual., Cornigliano S.p.A., Genova). *Metallurg. Ital.*, 1958, **50** (8), 325–328.—The zinc is cast into small discs and subjected to an arc-like discharge with a graphite counter-electrode. Traces of Al and Pb in the range 0·01 to 0·30% and 0·05 to 0·60%, respectively, are determined by using the 3018·35-A line of Zn as an internal standard. Coeff. of variation of 2·7 and 2·3% are found. The preparation of standards is described.

J. H. WATON

2085. **Dichrom[atom]etric determination of mercury(I).** G. Jagga Rao and K. Bhaskara Rao (Dept. of Chem., Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1959, **169** (4), 247–248 (in English).—Titration of Hg^I with $K_2Cr_2O_7$ is possible in $8 N$ HCl in the presence of ICl, which acts as catalyst and end-point indicator. The accuracy for the determination of 0·1 to 0·6 millimole of Hg is within $\pm 0\cdot2\%$. *Procedure*—To the $Hg_2(NO_3)_2$ sample add sufficient aq. HCl to keep the final acidity at $8 N$, $0\cdot02 M$ ICl (5 ml) and H_2O (to 50 ml). Cool, add CCl_4 (5 ml), and titrate with standard $K_2Cr_2O_7$ soln. to the ICl end-point; during the titration the Hg_2Cl_2 re-dissolves, and at the end-point the purple colour of the organic phase is discharged. J. P. STERN

2086. **Volumetric determination of bivalent mercury in the presence of thiofluorescein as indicator.** M. Wroński (Inst. Chem. Technol., Univ., Łodz, Poland). *Z. anal. Chem.*, 1959, **169** (5), 351–355 (in German).—Thiofluorescein has a blue colour in alkaline soln. which is discharged by Hg^{2+}, Ag^+, Cu^{2+} and Tl^+. Certain reagents, notably thioglycollic acid, will restore the blue colour, and this is the basis of the proposed method. The test soln., in a 250-ml conical flask, is made alkaline with N aq. NH_3, 1 ml of 0·02% thiofluorescein soln. [in a buffer soln. containing aq. NH_3 (2% w/v) and NH_4Cl (2% w/v)] is added, the soln. is diluted to about 100 ml and $0\cdot1 N$ to $0\cdot002 N$ thioglycollic acid is added to the first permanent blue colour. The interference of heavy metals can be overcome by the addition of EDTA. The accuracy is better than $\pm 2\%$. Micro quantities of Hg^{II} can be determined with an accuracy of 2% to 8% by a direct titration with diluted thiofluorescein soln. The method is applicable directly to organic compounds of the type RHgOH, while other organic compounds can be determined after preliminary decomposition with HNO_3 and H_2SO_4. H. M.

2087. **Colorimetric determination of boron in zirconium hydride.** G. R. Waterbury and C. F. Metz (Los Alamos Sci. Lab., N. Mex.). *U.S. Atomic Energy Comm.*, Rep. LA-1844, July, 1954. Decl. Sept., 1959. 35 pp.—The quant. determination of sub-microgram amounts of B in zirconium hydride is described. Dissolution of the sample in H_2SO_4 or HF and subsequent distillation of methyl borate, without loss of B or contamination, is shown to be possible. By using the curcumin colour reaction, amounts of B as low as 0·01 μg can be detected. The method should apply equally well to zirconium metal and compounds, provided that they can be dissolved under conditions that do not result in a loss of B and that the resulting soln. will permit the quant. removal of B by distillation as methyl borate. NUCL. SCI. ABSTR.

2088. **Complexometric determination of boron in the presence of barium.** O. Borchert (Lessingstr. 14, Schönebeck/Elbe, Germany). *Talanta*, 1959,

2 (4), 387–389 (in German).—Procedures are given for the determination of milligram amounts of B in salts for heat-treatment baths for steel. When Ba is absent from the sample, the B is pptd. with the reagent soln. (containing $BaCl_2$, NH_4Cl, aq. NH_3 and tartaric acid) and the excess of Ba in the filtrate is titrated directly with 0·01 to 0·1 M EDTA (disodium salt), with phthalein purple as indicator. For Ba-containing samples all the Ba is first complexed with EDTA and the excess of EDTA is then back-titrated with 0·01 to 0·1 M $MgCl_2$, with a mixed indicator [Tropaeolin OO (C.I. Acid Orange 5), Eriochrome black T and NaCl]. This titration is possible because the Ba in the EDTA complex cannot be displaced by Mg, which forms a very stable complex with the indicator. The end-point is the change from blue - green through grey to red. The aliquot of the sample soln. should contain 0·1 to 3 mg of B per 10 ml. The error is from $+0·12$ to $-1·9\%$ for 1 to 16 mg of $Na_2B_4O_7$ or 0·1 to 2 mg of B. (*Cf.* Ballczo and Doppler, *Mikrochim. Acta*, 1954, 403; 1956, 734.)

W. J. BAKER

2089. Integrating monitor for detecting low concentrations of gaseous boron hydrides in air. G. R. Fristrom, L. Bennett and W. G. Berl (Appl. Physics Lab., Johns Hopkins Univ., Silver Spring, Md., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1696–1697.—In the monitor described, the boron hydride vapours are quant. converted by burning into boric oxide, which is then determined colorimetrically with carmine at 585 mμ. In addition to monitoring over long periods, relatively rapid determinations of hydrides in the 1 p.p.m. concn. range can be made. The apparatus is applicable to all volatile boron derivatives that can react with excess of air to form boric oxide. K. A. PROCTOR

2090. Determination of metal ions with hexa-amminocobaltic chloride and ammonium fluoride. V. Gravimetric determination of aluminium. Yoshimasa Takashima (Chem. Dept., Fac. of Sci., Kyushu Univ., Hakozaki, Fukuoka). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (6), 619–622. —Aluminium (<20 mg) forms an insol. complex $[Co(NH_3)_6AlF_6]$ in the presence of NH_4F (>8 times the amount of Al) and an excess of hexa-amminocobaltic chloride (**I**). The ppt. is colloidal at pH <8, but can be washed with water and dried (110°, 1 hr.) to constant wt. when produced at pH 9. Scandium, Tl^{III} and Ga form ppt., but Tl^{I}, Cu, Be (<1 mg per $<2·5$ mg of Al), Cd, Zn, Ni, Ti and Zr do not.

VI. Determination of gallium. Yoshimasa Takashima. *Ibid.*, 1959, **80** (6), 622–624.—A quant. pptn. of Ga (<10 mg) is effected at pH 3·5 to 6 in the presence of F$^-$ (>5 times the wt. of Ga) and a small excess of **I**. The ppt. is washed with water and dried at 110°. Interference results from In and Tl^{III}, but not from Mn, Zn, Ni, Tl^{I} or Ti^{IV}.

VII. Radiometric determination of aluminium and gallium. Yoshimasa Takashima. *Ibid.*, 1959, **80** (6), 624–625.—The procedure described above is carried out on a micro scale with **I** labelled with ^{60}Co and the ppt. is counted with a Geiger - Müller counter. The calibration curve is linear for 5 to 20 μg and 50 to 250 μg of Ga and Al, respectively, under the given conditions. K. SAITO

2091. Ion-exchange separation and colorimetric determination of aluminium. Chen-Chiang Liao. *Acta Chim. Sinica*, 1959, **25** (3), 152–159.—The colorimetric determination of Al by means of

Eriochrome cyanine R (C.I. Mordant Blue **3**), after the separation of interfering ions with Zeolite FF (chloride form), is further examined. Procedures for the determination of Al in alloy steel, copper - aluminium alloys and iron ore have been devised. The determination of Al in alloy steel or in copper - aluminium alloys takes 0·5 hr. and its determination in iron ore takes 1 hr. SCI. ABSTR. CHINA

2092. Determination of aluminium in solutions containing fluoride ion by means of 8-hydroxyquinoline. I. V. Tananaev and A. D. Vinogradova (N. S. Kurnakov Inst. of Gen. and Inorg. Chem., Acad. Sci. USSR, Moscow). *Zhur. Anal. Khim.*, 1959, **14** (4), 487–488.—In the determination of Al with 8-hydroxyquinoline in acetate buffer soln. the interference caused by the presence of F$^-$ is avoided by the addition of a beryllium salt in an amount that provides a molar ratio of Be to Al of 1:1. Larger amounts of Be do not interfere.

C. D. KOPKIN

2093. Determination of aluminium in high-temperature alloys. K. L. Cheng and F. J. Warmuth (Metals Div., Kelsey-Hayes Co., New Hartford, N.Y.). *Chemist Analyst*, 1959, **48** (3), 74–76.— Detailed procedures are given for the determination of Al in alloys containing many interfering metals. Preliminary separations are carried out with a mercury cathode to remove Ni, Co, Fe and part of the Mo and Cr, followed by pptn. with cupferron to remove the remainder of the Mo together with Zr, Ti, Ta and Nb; the residual Cr is volatilised as chromyl chloride. The Al is then determined by EDTA titration, with 1-(2-pyridylazo)-2-naphthol as indicator. G. S. ROBERTS

2094. Spot-test identification of wrought aluminium and magnesium alloys. J. A. Catoggio (Dirección Nacional de Quím., Buenos Aires, Argentina). *Chemist Analyst*, 1959, **48** (3), 58–65.— Two systematic schemes of identification are given, one for magnesium-base and one for aluminium-base alloys. The schemes depend upon the application of spots of reagent to the cleaned alloy surface. Identification may take as long as an hour, but is essentially non-destructive and can be carried out outside the laboratory. G. S. ROBERTS

2095. Colorimetric determination of aluminium in acid solutions of phosphate rock. W. A. Jackson (Fertilizer Invest. Res. Branch, Soil and Water Conservation Div., U.S. Dept. of Agric., Beltsville, Md.). *J. Agric. Food Chem.*, 1959, **7** (9), 628–630.— Iron interferes in the colorimetric determination of Al (as the complex with Na alizarinsulphonate) in acid soln. of phosphate rock. The interference is avoided by converting Fe into the cupferron complex, and extracting it with $CHCl_3$. Aluminium was determined in synthetic samples with ratios of Fe_2O_3 to Al_2O_3 of up to $>7:1$; the results agreed well with those of gravimetric determinations.

M. D. ANDERSON

2096. Further contribution to the analysis of aluminium oxide. E. Krejzová, J. Kruml and L. Plocek (Res. Inst. Electrotech. Ceramics, Hradec Králové, Czechoslovakia). *Sklář a Keram.*, 1959, **9** (8), 244.—*Procedure for SiO_2*—Fuse the sample (2 g) with Na_2CO_3 - $Na_2B_4O_7$ (5:2) (15 g), dissolve in HCl (1:5) (150 to 200 ml) and evaporate carefully to dryness. Dry at 130°, moisten with conc. HCl and dilute with hot H_2O to about 200 ml and set aside on the water bath till dissolution is complete,

Filter off and wash the silicic acid, and determine SiO_2 in the usual way. *Procedure for CaO and MgO*—Dilute the filtrate to 500 ml, and dilute 200 ml of this soln. to 1·5 litres; add triethanolamine (20 ml) and 3 N NaOH (80 ml) to adjust the pH to 11 to 13. Add murexide indicator and titrate with 0·01 M EDTA (disodium salt) (I) to a violet endpoint. Dilute a second 200 ml to 1·5 litres, add NH_4Cl soln. (20%), triethanolamine (120 ml), buffer soln. (50 g of NH_4Cl and 350 ml of conc. aq. NH_3 per litre) (150 ml), the vol. of I consumed in the previous titration, KCN soln. (5%) (15 ml) and Eriochrome black T indicator, and titrate with 0·01 M I till blue. Ferric oxide and TiO_2 are determined colorimetrically (with 2:2'-dipyridyl and H_2O_2, respectively) and Na_2O and K_2O flamephotometrically.   J. ZÝKA

2097. Polarographic determination of mixtures of aluminium and gallium. B. A. Cooney and J. H. Saylor (Dept. of Chem., Duke Univ., Durham, N.C., U.S.A.). *Anal. Chim. Acta*, 1959, 21 (3), 276–282 (in English).—The metals are polarographed as the chelate complexes of 2-hydroxy-5-sulphophenylazoresorcinol (C.I. Mordant Red 5). The supporting electrolyte is 0·1 M KCl buffered to pH 5·53 and containing gelatin as maximum suppressor. Aluminium gives waves at $-0·34$ and $-0·54$ V and Ga at $-0·34$, $-0·54$ and $-1·05$ V *vs.* the S.C.E., respectively. The concn. of Ga is calculated from the wave at $-1·05$ V and the concn. of Al from the wave at $-0·54$ V after correcting for the Ga present. The procedure has a precision of $\pm$ 6% for Al and $\pm$7·8% for Ga for mixtures of Al and Ga in the range of 1:1 to 5:1. Zinc, Mn^{II}, Ce^{IV}, Fe^{III}, V^{IV} and Ni interfere; Cd and Pb interfere with the determination of Al but not of Ga. By using an ether-extraction step, mixtures of Al and Ga in a ratio of 100:1 can be analysed for Ga but not for Al.   W. T. CARTER

2098. Spectrochemical analysis of high-purity gallium. E. B. Owens (Lincoln Lab., M.I.T., Lexington, Mass., U.S.A.). *Appl. Spectroscopy*, 1959, 13 (4), 105–108.—Gallium is separated from its impurities by extraction of $HGaCl_4$ with di*iso*propyl ether from HCl soln. After extraction, the vol. of the acid phase is reduced and a measured portion, to which Mo is added as an internal standard, is dried on a waxed graphite electrode. The spectra are excited with a condensed spark, and the max. sensitivities reported are—0·03 p.p.m. of Al and Ca, 0·06 p.p.m. of Cr, Pb and Ni, 0·015 p.p.m. of Cu and Mg, and 0·12 p.p.m. of Zn; coeff. of variation range from $\pm$ 3·6% for Cu to $\pm$ 14·8% for Pb. The method should be applicable to the analysis of any high-purity material which can be separated by solvent extraction from its impurities.
                                      K. A. PROCTOR

2099. Separation and determination of gallium, indium and thallium with hexa-amminocobaltic chloride. Yoshimasa Takashima (Chem. Dept., Fac. of Sci., Kyushu Univ., Hakozaki, Fukuoka). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, 80 (6), 626–628.—Whilst In and Tl^I do not form ppt. with hexa-amminocobaltic chloride (I) and NH_4F, Tl^{III} and Ga are quant. pptd. with these reagents at pH $<0·6$ and 3·5 to 6·0, respectively. A sample soln. containing <10 mg each of Ga, In and Tl^I is dissolved in 2·5 N HCl and the In is pptd. with I in the absence of F−, washed with water, filtered off and weighed. The Tl in the filtrate is oxidised with aq. Br soln. and pptd. with I in 0·5 N HCl. The Ga is

pptd. with I and NH_4F at pH 5 and weighed. This method is extended to the μg scale by the use of I labelled with ⁶⁰Co (*Anal. Abstr.*, 1960, 7, 2090).
                                      K. SAITO

2100. Use of the method of additions in the spectrographic analysis of ores for indium and germanium. Ya. D. Raĭkhbaum and E. S. Kostyukova (Irkutsk State Inst. of Rare Metals). *Zavod. Lab.*, 1959, 25 (8), 961–963.—The samples and standards are mixed with equal parts of a buffer mixture of C and $NaHCO_3$ (1:1). Further quantities of the samples are also mixed with the same buffer mixture but containing also a known amount of the element to be determined. The mixtures are excited on a rotating electrode and the spectra of both mixtures of the samples and the standards are obtained on one plate. The concn. of In and Ge are calculated from the line intensities and a calibration curve, and the use of a formula. The method is applied to the determination of small amounts (0·001 to 0·01%) of In and Ge in various concentrates, ores, coal, etc.   G. S. SMITH

2101. Iodimetric determination of thallium. Hsi-Yu Chang and Yüng-Huei Yeh. *Acta Sci. Nat. Univ. Pekin.*, 1959, (3), 241–245.—The semi-micro method of Sill and Peterson for the iodimetric determination of Tl has been extended to the macro scale by the adoption of either of the following procedures. Satisfactory results may be obtained in each case. (*i*) The addition of 1% gum acacia soln. (15 to 40 ml) and 95% ethanol (5 to 20 ml) to the soln. to be titrated improves the accuracy of the determination when the concn. of both Tl^+ and $Na_2S_2O_3$ are in the range 0·005 to 0·05 N. (*ii*) The addition of KBr is more effective. When the concn. of both Tl^+ and $Na_2S_2O_3$ are in the range 0·05 to 0·1 N, the optimum conditions have been found to be—acid concn. 0·2 to 1·0 N in H_2SO_4 (final acid concn.); KBr concn. 1·0 to 1·5 M (approx. 8 to 12 g of KBr); KI concn. 0·0036 to 0·0055 M (approx. 0·04 to 0·06 g of KI); the final vol. of the soln. should be $\simeq 70$ ml.   SCI. ABSTR. CHINA

2102. Photometric determination of thallium with tetramethyldiaminodiphenylantipyrinylmethanol. A. I. Busev and V. G. Tiptsova. *Nauch. Dokl. Vÿssh. Shkolÿ. Khim. i Khim. Tekhnol.*, 1959, (1), 105–107; *Ref. Zhur., Khim.*, 1959, (20), Abstr. No. 71,235.—It is shown that Tl^{3+} in the presence of Cl^- and Br^- form a blue - violet ppt. with tetramethyldiamino-diphenylantipyrinylmethanol (I) which is easily extracted by diethyl ether or benzene and its homologues with the formation of an emerald-green soln. I is not extracted by the solvents mentioned but is extracted by $CHCl_3$, amyl acetate and dichloroethane. The absorption of the coloured extract is a max. at 638 mμ; the mol. coeff. of extinction at this wavelength is 68,000. The extinction is practically constant in the range 0·1 to 1·0 N HCl. In the determination of Tl^+ by means of I, first oxidise Tl with chlorine water, then add 2 N HCl (5 ml) to the soln. (10 ml, containing $\simeq 10$ μg of Tl) and heat until all the Cl has been driven off (test with starch - iodide paper); cool the soln., transfer it to a separating-funnel with N HCl, add 0·5% I soln. (1 to 2 ml) followed by 10 ml of benzene - CCl_4 (2:3), and extract for 1 min.; set aside for 5 min., and measure the extinction of the lower layer at 638 mμ. The method can be used for the determination of 1 to 20 μg of Tl in 10 ml of soln.; the error is 10 to 15%. The determination is unaffected by the presence of $CdCl_4^{4-}$, $BiCl_4^-$,

MoO_4^{2-} and WO_4^{2-} (which form coloured ppt. with I that are not extracted by the solvents mentioned); Fe^{3+} and Cu^{2+} also have no effect; Zn, Hg, Sb and Sn interfere.　　　　K. R. C.

2103. Spectrographic analysis of high-purity thallium. D. M. Shvarts and I. S. Nilova ("Gipronikel' " Inst.). *Zavod. Lab.*, 1959, **25** (8), 949–952.—The sample of metal is dissolved in HNO_3, the soln. is evaporated, and the dry $TlNO_3$ is analysed directly or after enrichment of the impurities. In the direct analysis, 15 elements in concn. from 10^{-3} to 10^{-4} % can be determined in a d.c. arc (6 amp., 400 V) between carbon electrodes if complete evaporation is carried out. After enrichment of the impurities the $TlNO_3$ is evaporated off at 300° in a special oven, and the residue is excited in a d.c. arc. This method is more sensitive.
　　　　　　　　　　　　　　　　G. S. SMITH

2104. Volumetric determination of thallium in sodium iodide crystals. M. J. Toogood (Ekco Electronics Ltd., Southend-on-Sea, Essex, England). *Analyst*, 1959, **84**, 618.—For routine determinations of Tl ($\simeq 0.05\%$) in single crystals of sodium iodide intended for measurement of gamma radiation, the sample is dissolved in a soln. of $0.02\ N$ iodine in 0·6% (w/v) NaI soln. and the mixture is extracted with *n*-butanol. The extract is boiled with 5% (w/v) NaI soln. to remove *n*-butanol, and the aq. liquid is washed with $CHCl_3$ to remove iodoform. Residual $CHCl_3$ is removed by boiling and the soln. is titrated with $0.05\ N\ Na_2S_2O_3$ until a faint turbidity indicates incipient pptn. of thallous iodide. The titration is then continued, with starch as indicator. This second titration measures the amount of iodine needed to keep the thallous iodide in solution and hence the wt. of Tl present can be calculated, the factor being 0·00425. None of the common acidic ions interferes. Reducing agents must be completely oxidised before the titration.　　　　A. O. JONES

2105. Thermo-spectrophotometric determination of the rare earths in the presence of thorium. V. I. Kuznetsov and T. V. Petrova (V.I. Vernadskii Inst. of Geochem. and Anal. Chem., Acad. Sci. USSR, Moscow). *Zhur. Anal. Khim.*, 1959, **14** (4), 404–410.—Thermo-spectrophotometry is spectrophotometry of a soln. carried out at different temperatures. At pH 3·6 to 3·8 the extinction of the arsenazo complex of Th at 576 $m\mu$ is practically the same at 80° as at 20°, while for the rare-earth metals it is greater at the higher temp., *e.g.*, for La 1·9 times, and for Yb 2·9 times. Metals other than alkali metals, and masking anions, should be absent. To construct a calibration curve, in each of five 25-ml flasks place $Th(NO_3)_4$ soln. ($\equiv 0.23$ mg of ThO_2), add soln. of the nitrates of the rare-earth elements ($\equiv 0$, 0·005, 0·010, 0·020 and 0·040 mg of R_2O_3), $\simeq 5$ ml of water, 2 drops of a 0·1% phenolphthalein soln., 0·1 N NaOH till the soln. is red, 2 drops of 0·05 N HCl, a further 0·016 ml of 0·05 N HCl, 8·0 ml of a 0·3% aq. soln. of arsenazo and 0·4 ml of buffer soln. (pH 3·7) (dissolve 4·72 g of chloroacetic acid in 10 ml of water, add 21·8 ml of 2 N NaOH in small portions with stirring, and dilute to 50 ml), and make up to 25 ml. Measure the extinction at 20° and 80° in a 40-mm cell at 576 $m\mu$; construct the calibration curve from the differences in the readings. To prepare the sample soln., dissolve solid nitrates or chlorides ($\equiv \simeq 10$ mg of total oxides) in 0·1 $N\ HNO_3$ and make up to 100 ml with this acid. With oxalates, evaporate to dry-

ness with 10 ml each of conc. HCl, $HClO_4$ and HNO_3, then ignite till no more white fumes are evolved; dissolve the residue in 10 ml of $N\ HNO_3$ and dilute to 100 ml. To determine the rare-earth elements, use an aliquot of the soln. ($\equiv 0.23$ mg of ThO_2) and treat as for the construction of the calibration curve. To determine Th, to 1 ml of the soln. add about 5 ml of water, 2 drops of 0·1% phenolphthalein soln., 0·1 N NaOH till the soln. is red, 0·1 N HCl till colourless, a further 2 ml of 0·1 N HCl, 4 ml of 0·3% arsenazo soln. and 2 ml of buffer soln. (pH 2·0) (dissolve 4·72 g of chloroacetic acid in 10 ml of water, add 6·12 ml of 2 N NaOH, with stirring, and dilute to 50 ml), and make up to 25 ml. Measure the extinction in a 10-mm cell at 576 $m\mu$. The thorium content of the 100-ml sample soln. is then $E/3.70$ mg of ThO_2 per ml, where E is the extinction, and 3·70 is the extinction for 1·00 mg of ThO_2 in 25 ml. The method can be used to determine 1·2% of rare-earth elements (as oxides) in the total oxides. With 1·2% of R_2O_3 in ThO_2 the error is 7%, the error decreasing to 1% with 15% of R_2O_3.　　　　C. D. KOPKIN

2106. Spectrographic analysis of preparations of the rare-earth cerium group for samarium. V. L. Kustas and G. V. Lazebnaya. *Zavod. Lab.*, 1959, **25** (8), 958–959.—The sample soln. (0·05 to 25%) (one drop) is placed on the end of a graphite electrode, which has previously been treated with a 3% soln. of polystyrene in benzene and dried, and the sample soln. is dried at 100°. The residue is excited in an a.c. arc (10 amp.).　　G. S. SMITH

2107. Determination of small amounts of rare earths in praseodymium, neodymium, samarium, terbium, dysprosium, holmium, erbium and thulium preparations. T. I. Grishina (M. V. Lomonosov Moscow State Univ.). *Zhur. Anal. Khim.*, 1959, **14** (4), 427–430.—One drop of a 5% soln. (as metal) of the chlorides of the rare-earth elements in 1% HCl is placed on the flat end of a copper or carbon electrode (coated with a film of polystyrene) and dried. The spectra are excited in an a.c. arc at 8 amp. and 220 V. For the pairs of metals studied, the analytical lines (in A), determinable concn. (%) and electrodes are—Y in terbium, Y 3774·33 and Tb 3761·95 A; 0·02 to 0·2; copper; Gd in terbium, Gd 3768·41 and Tb 3761·95; 0·02 to 1; copper; Y in dysprosium, Y 3774·33 and Dy 3777·99; 0·007 to 0·2; copper; Dy in holmium, Dy 4211·72 and Ho 4219·13; 0·02 to 1; copper; Ho in erbium, Ho 4103·84 and Er 4104·01; 0·02 to 1; copper; Tm in erbium, Tm 3362·61 and Er 3369·22; 0·005 to 1; carbon; Yb in thulium, Yb 3289·37 and Tm 3291·00; 0·05 to 1; carbon (for a 0·05% soln. of Tm in 1% HCl); or Yb 3987·99 and Tm 3926·74; 0·005 to 0·2; copper; Er in thulium, Er 3906·32 and Tm 3912·90; 0·02 to 1; copper; La in praseodymium, La 3337·49 and Pr 3340·53; 0·005 to 0·1; carbon; Nd in praseodymium, Nd 4358·17 and Pr 4356·67; 0·1 to 1; carbon; Sm in neodymium, Sm 4433·87 and Nd 4436·68; 0·05 to 1; carbon; Pr in neodymium, Pr 4408·84 and Nd 4419·62; 0·1 to 1; carbon; Nd in samarium, Nd 4303·57 and Sm 4300·16; 0·02 to 1; carbon; Eu in samarium, Eu 3819·66 and Sm 3818·35; 0·005 to 0·2; copper. Calibration curves are constructed on the co-ordinates log (relative intensity) *vs.* log [concn. (%) of impurity metal]; standards are prepared from the purest possible rare-earth materials obtained by ion-exchange chromatography; the original content of impurity is determined by the method of additions.　　　　C. D. KOPKIN

2108. Chemical studies on radioactive indicators. XX. Separation of lanthanum from a barium - lanthanum transient equilibrium mixture with a mercury cathode. Masayoshi Ishibashi, Taitiro Fujinaga, Atsuyoshi Saito and Kunzo Masuda (Fac. of Sci., Kyoto Univ., Sakyo-ku, Kyoto). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (5), 490–492.—Hildebrand's double cell was applied to the separation of ^{140}La from ^{140}Ba with a mercury cathode; with a terminal potential of 10 to 15 V, La is found near the surface of the mercury in the inner cell and not on that in the outer cell. The La is readily extracted with dil. HNO_3. The yield is ≃30% after electrolysis for 1 hr.   K. Saito

2109. Analysis of yttrium metal and yttrium oxide. I. Copper, nickel, iron and molybdenum. J. O. Hibbits, W. F. Davis and M. R. Menke (General Electric Co., Aircraft Nucl. Propulsion Dept., Cincinnati, Ohio). *U.S. Atomic Energy Comm.*, Rep. APEX-519, Sept., 1959. 22 pp.—The Cu, Ni, Fe and Mo were separated from yttrium by liquid - liquid extraction. No difficulties were encountered in the determination of Cu, Ni or Fe. Some difficulty was experienced with the determination of Mo when large amounts of Cu were also present. Several washings were necessary to eliminate the turbidity caused by the pptn. of Cu as cuprous thiocyanate.   Nucl. Sci. Abstr.

2110. Sequential separation of some actinide elements by anion exchange. F. P. Roberts and F. P. Brauer (General Electric Co., Hanford Atomic Products Operation, Richland, Wash.). *U.S. Atomic Energy Comm.*, Rep. HW-60552, June, 1959. 9 pp.—Methods are described by which trace amounts of several actinide elements can be separated. Use is made of the large differences in distribution coefficients, so that careful chromatographic techniques are not necessary. Small columns are used, allowing the desired constituent to be obtained in 10 ml or less. Americium, Pu and Np are separated (in that order) by sequential elution from columns of Dowex-1 resin with 8 M HNO_3, 0·02 M ferrous sulphamate in 4·5 M HNO_3, and 0·001 M Ce(SO$_4$)$_2$ in 0·25 M HNO_3. Americium, Th, Pu and Np are separated sequentially (in that order) by elution with 8 M HNO_3, 12 M HCl, 12 M HCl - 0·1 M NH$_4$I, and 4 M HCl. Protactinium and U follow the Am in both separations. The methods described are characterised by a low degree of cross-contamination. Yields are >95%.   Nucl. Sci. Abstr.

2111. Method for determining carbon-14 by combustion using calcium carbonate. E. C. S. Little (Dept. of Agric., Univ. of Oxford, England). *Nature*, 1959, **184** (Suppl. 12), 900.—When there is adequate carbon available (>10 mg for a 2·4-cm-diameter end counting window) the use of CaCl$_2$ instead of BaCl$_2$ for pptg. the carbonate gives an increased counting rate. The ppt. formed with CaCl$_2$ tend to be gelatinous and to give uneven preparations, but this can be avoided by the use of MgCl$_2$ with the CaCl$_2$ in the ratio of 1:2. The ppt. from the mixed chlorides are probably slightly contaminated with magnesium hydroxide.   K. A. Proctor

2112. Measurement of carbon-14 with a gas counter. F. Dutka, O. Orient and D. Gál (Isotope Lab., Res. Inst. for Soil Sci. and Agric. Chem., Hung. Acad. Sci., Budapest). *Magyar Kém. Foly.*, 1959, **65** (8), 328–329.—The ^{14}C to be measured is introduced into the counter as CO$_2$, or more rarely as methane, ethane or acetylene. The discharge is quenched by means of ethanol (carbon disulphide is less convenient) and an external quenching circuit of the Neher - Harper type. The plateau obtained covers a range of 240 V and the slope is <0·5%. The background of the unprotected counter was found to be 50 impulses per minute. The memory effect is negligible.   A. G. Peto

2113. Use of tertiary alkyl primary C$_{12}$ to C$_{14}$ amines for the assay of ^{14}CO$_2$ by liquid scintillation counting. R. A. Oppermann, R. F. Nystrom, W. O. Nelson and R. E. Brown (Dept. of Dairy Sci., Coll. of Agric., Univ. of Illinois, Urbana). *Int. J. Appl. Radiation and Isotopes*, 1959, **7**, 38–42.—Distillation of Primene 81-R (a mixture of tertiary amines with an average mol. wt. of 191) *in vacuo* at 160° gives a clear reagent, of which a 0·25 M soln. in methanol has been used to trap ^{14}CO$_2$. The concn. of CO$_2$ was determined by titrating an aliquot of the soln. with standard alkali. A second aliquot (1 ml) was added to 13 ml of a 0·3% soln. of 2:5-diphenyloxazole in toluene and the mixture was then counted. The Primene - CO$_2$ complex and the scintillation mixture were stable for 14 days at 25° and −8°, respectively. The stability of the scintillation mixture at 25° can be increased by using a higher concn. of the amines or by sealing it in a vessel.   G. J. Hunter

2114. Photometric determination of traces of carbon monoxide in the presence of hydrogen. G. Ciuhandu and G. Krall (Inst. Hygiene, Timisoara, Romania). *Rev. Chim., Bucharest*, 1959, **10** (9), 531–534.—The method is based on measurement of the extinction of the product obtained by treatment of the mixed gases with an alkaline soln. of Ag p-sulphamoylbenzoate (**I**), prepared by mixing 1 vol. of 0·1 M Na p-sulphamoylbenzoate with 1 vol. of 0·1 M AgNO$_3$, and dissolving the ppt. in 0·5 vol. of 1 M NaOH. Standard curves are prepared with varied percentages of H and CO. For the determination the concn. of H must be adjusted to that of a standard curve, since the relationship between extinction (at 420 mμ) and concn. of H is not linear. Ammonia, H$_2$S, aldehydes and thiols interfere, and must be removed. *Procedure*—The mixture, diluted with air to give a concn. corresponding to the standard curve, is introduced into a 100-ml flask by displacement of water, and set aside for 30 min. at 18° to 20°; 10 ml of **I** soln. is introduced, and the mixture is set aside for 22 hr. at 20°. The extinction of the soln. is then measured, and the percentage of CO obtained from the standard curve. The results are consistent for concn. of H up to 20%, and concn. of CO from 0·001 to 0·4%, provided that the stated conditions are strictly adhered to and that the same photometer is used for sample and standards. A description of the apparatus is given.   H. Sher

2115. Determination of small amounts of carbon monoxide in hydrogen- and methane-containing gases. J. Sverak (Hauptlab. der Österreich. Stickstoffwerke A.-G., Linz, Austria). *Mikrochim. Acta*, 1959, (6), 908–915 (in German).—The method is based on the combustion of CO to CO$_2$, followed by potentiometric titration of the CO$_2$. Oxygen serves both as a reactant and carrier gas. Copper oxide, which acts rather as a heterogeneous catalyst, is constantly regenerated and this enables gases

containing even large amounts of H to be continuously analysed. By maintaining a furnace temp. of $285° \pm 5°$ any methane present in the sample will not be burned. As little as 0·03% of CO_2 in 100 ml of gas can be determined with a relative error $\not> 3\%$. A determination requires from 20 to 30 min. The potentiometric titration readily lends itself to automation.

<div align="right">D. F. PHILLIPS</div>

2116. **Spectrographic determination of impurities in high-purity carbon and graphite.** A. A. Demidov and L. B. Gorbunova. *Zavod. Lab.*, 1959, **25** (8), 956–957.—The finely ground sample (5 g) is mixed with 100 mg of BeO and ignited at 800° for 12 to 15 hr., the BeO serving as a collector for small amounts (10^{-3} to $10^{-5}\%$) of Al, Mg, Si, Fe and Ca. The BeO (with the impurities) is excited on a carbon electrode in a d.c. arc.   G. S. SMITH

2117. **Studies in the determination of silica. II. Separation of molybdophosphate from α-molybdosilicate.** L. H. Andersson (Dept. of Anal. Chem., Inst. of Chem., Univ., Uppsala, Sweden). *Acta Chem. Scand.*, 1959, **13** (9), 1743–1752 (in English).—Data on the variation in extinction of molybdophosphate with pH and $MoO_4{}^{2-}$ concn. at 400 mμ are given and a theoretical treatment of the extraction of molybdophosphoric acid (**I**) or α-molybdosilicic acid (**II**) is presented. Two solvent systems (n-butanol and n-butanol-benzene) and the effect of salts in the aq. phase are discussed; the concn. of $SO_4{}^{2-}$ must be low to prevent co-extraction of **II**. With the second system, a molar fraction of 0·5 for n-butanol makes the separation of **I** and **II** possible; the influence of temp. between 18° and 24° is negligible. *Determination of phosphorus*—Adjust the aq. soln. ($0·2 \times 10^{-3} M$ in P and 0·02 M in Na_2MoO_4) to pH 1·25 with HCl and extract with 10 ml of the organic solvent. Repeat the extraction, dilute the extracts to a known vol. with the solvent and read the extinction at 400 or 310 mμ.

<div align="right">P. D. PARR-RICHARD</div>

2118. **Rapid determination of silica and iron in chrome ores.** H. Grubitsch (Inst. für anorg.-chem. Technol. u. anal. Chem., Tech. Hochsch., Graz, Austria). *Radex Rdsch.*, 1959, (1), 460–461.—*Procedure*—Fuse the sample (0·5 to 1 g) with Na_2O_2 and NaOH in a nickel crucible. Dissolve the melt in 20 ml of H_2O and wash out the crucible with a little H_2O. To the soln. in a beaker add 60 ml of dil. H_2SO_4 (1:1), heat until clear and add 1 ml of 30% H_2O_2 soln. Heat strongly until gelatinous SiO_2 separates on the sides of the beaker (15 to 20 min.), dilute to 100 ml and cool to 65°. Add, with vigorous stirring, hot filtered 2% gelatin soln. (5 ml), set aside for 1 hr. and filter cold. Wash the ppt. 6 times with a soln. containing H_2O (500 ml), H_2SO_4 (10 ml) and 2% gelatin soln. (10 ml); (reserve the filtrate for the determination of Fe); then 3 times with a similar soln. containing HNO_3 (10 ml) in place of H_2SO_4; reject these washings. The pptd. SiO_2 is then determined in the usual way. The difference between the results and those by a standard method is $\not> 1·4\%$. The procedure occupies 5 hr. Determine Fe in the filtrate (diluted to 300 ml) by the cadmium reductor method, with ferroin as indicator for the $KMnO_4$ titration.

<div align="right">P. D. PARR-RICHARD</div>

2119. **Use of trihydroxyfluorone derivatives in colorimetric analysis. Reagents for germanium.** V. A. Nazarenko and N. V. Lebedeva (Inst. Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1959, **25** (8), 899–903.—The properties of a number of complexes formed by derivatives of trihydroxyfluorone with Ge are studied. Disulphophenylfluorone is useful in weakly acid soln., but phenylfluorone or nitrophenylfluorone is necessary when Ge has to be determined in strongly acid soln. The use of p-dimethylaminophenylfluorone for determining Ge is not recommended, although it is suitable for Ta.   G. S. SMITH

2120. **Spectrophotometric studies on organometallic complexes used in analytical chemistry. V. The reaction of organic reagents with germanium.** Takuji Kanno (Res. Inst., Mineral Dressing and Metallurgy, Tohoku Univ., Nagamachi, Sendai). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (7), 757–760.—The fact that compounds containing a carbonyl and an hydroxyl group *ortho* to one another react with Ge in acid medium and that those containing two hydroxyl groups *ortho* to one another react in neutral soln. (*Anal. Abstr.*, 1959, **6**, 95) was further demonstrated with cyanidin chloride, haematein and haematoxylin.   K. SAITO

2121. **Volumetric determination of germanium as catechol-germanic acid.** E. Wunderlich and E. Göhring (Unterharzer Berg- und Hüttenwerke G.m.b.H., Goslar, Germany). *Z. anal. Chem.*, 1959, **169** (5), 346–350 (in German).—Germanium forms a strongly acid complex with catechol which can be titrated with standard alkali. Tervalent As does not interfere, but if Sb^{III}, Sn^{IV}, Fe^{III} or boric acid is present the Ge is separated by distillation as $GeCl_4$ from HCl soln. The distillate is made up to a suitable volume, and an aliquot containing 20 to 100 mg of Ge is taken. The soln. is carefully neutralised with 10% carbonate-free alkali to methyl red, and is diluted to 250 ml. After the temp. of the soln. has been adjusted to 20° the pH is brought to 5·0, 3 g of catechol is added and 0·1 N alkali (previously standardised with pure Ge soln.) is added from a burette until the pH is again 5·0. The method gives results comparable with those of a gravimetric procedure.   H. M.

2122. **New luminescence method of microchemical analysis (crystallophosphor method). II. Detection of tin.** K. P. Stolyarov and N. N. Grigor'ev (A. A. Zhdanov Leningrad State Univ.). *Zhur. Anal. Khim.*, 1959, **14** (4), 491–492.—Tin is detected by means of the crystallophosphor KI.Sn. Place a drop of test soln. (0·001 ml) on a filter-paper by means of a capillary, and add to the centre of the spot a drop of KI soln. so that it completely covers the first spot. Dry the filter-paper and inspect under u.v. light. In the presence of considerable amounts of Sn a yellow luminescence is observed, surrounded by a red ring due to iodine; with small amounts of Sn a diffuse but marked luminescence on a red field is observed. If the luminescence is slight, the paper is re-dried and the radiation strengthened; too much heating is harmful, since the iodine formed absorbs u.v. light and weakens or destroys the luminescence. The detectable minimum is 0·02 μg of Sn at a limiting dilution of 1 in 5×10^5. In the presence of Hg, first use a drop of KI soln., then the test soln., and then KI soln. to dissolve the ppt. of mercury iodides. The influence of small amounts of Tl may be avoided by using light of wavelength 365 mμ; the luminescence due to Tl is then much weakened, the luminescence due to Sn being greenish yellow. With large

amounts of Tl, part at least of the Sn is separated by extraction before the test; Sb^{3+}, Bi^{3+}, As^{3+}, Ag^+ and Pb^{2+} may interfere.　　　C. D. KOPKIN

2123. Photometric determination of antimony and thallium in lead. C. L. Luke (Bell Telephone Laboratories Inc., Murray Hill, N.J., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1680–1682.—After being separated from Pb by co-pptn. with MnO_2 from HNO_3 soln., Sb and Tl are determined by photometric Rhodamine B procedures. The method is suitable for up to 15 μg of Sb and up to 20 μg of Tl, and a precision of ± 1 μg is reported.
　　　　　　　　　　　　　T. R. ANDREW

2124. The detection of titanium as the tetraphenylarsonium salt of polyhydric phenol chelates. L. Sommer (Inst. Anal. Chem., Univ., Brno, Czechoslovakia). *Z. anal. Chem.*, 1959, **169** (5), 342–346 (in German).—A number of polyhydric phenols give orange colours with Ti in the presence of tetraphenylarsonium chloride (**I**) in acid soln. The phenols preferred are protocatechualdehyde, 2:3-dihydroxynaphthalene-6-sulphonic acid, pyrogallol, catechol and 2:3:4-trihydroxybenzoic acid. To 1 to 2 drops of the test soln. in a semi-micro test-tube are added 1 ml of reagent soln. and about 20 mg of **I**. The tube is well shaken and 1 ml of buffer soln. of pH 2·7 and 1 ml of $CHCl_3$ are added. The tube is again shaken, and in the presence of Ti the $CHCl_3$ layer is coloured yellow to orange. A blank may be necessary. Interference can be expected from Zr^{IV}, Fe^{III}, U^{VI}, Nb^V, Ta^V, Mo^{VI} and V^V, but not from Ni^{II}, Co^{II}, Mn^{II}, Zn^{II}, Cd^{II}, Mg^{II}, Sr^{II}, Ca^{II}, Ba^{II}, Al^{III}, Th^{IV} and $WO_4{}^{2-}$.　　　H. M.

2125. Spectrometric determination of traces of titanium with a new specific reagent. P. M. Strocchi and P. Rebora (Fac. Engng, Univ. Bologna, Italy). *Z. anal. Chem.*, 1959, **169** (1), 1–10 (in German).—The dihydroxylamine salt of dihydroxymaleic acid (prep. described) is a specific reagent for the spectrometric determination of traces of titanium. To 10 ml of the sample soln. (acidified with HCl or H_2SO_4 to pH 2·40 $\pm$ 0·05) is added 7 ml of reagent soln. (0·2857 g in 100 ml of water at pH 6 to 7), and the vol. is made up to 25 ml. After a development time of 25 min., the extinction of the soln. is measured against a blank soln. containing no reagent, and compared with a calibration curve. The effects of concn. of reagent, hydrogen ion concn. and development time are considered. Vanadium, Fe, Cr, Mg, Ca, Sr, Ba, Cu, Ni and Si do not interfere nor, in limited quantities, do Al, Mn and F. As little as 0·025 μg of Ti per ml can be determined.
　　　　　　　　　　　　　S. M. MARSH

2126. Photometric determination of titanium in titanium borides with arsenazo. E. I. Nikitina. *Zhur. Anal. Khim.*, 1959, **14** (4), 431–433.—Dissolve 0·2 g of titanium boride, metallic titanium or titanium chromium boride in H_2SO_4 (1:1) (20 ml) with H_2O_2 (7 ml), heat to fumes of SO_3 and transfer the soln. to a 100-ml flask; filter off and wash any insol. residue, fuse it with $K_2S_2O_7$ and add the soln. of the melt to the main soln.; make up to volume. Dilute 5 ml to 200 ml, and to a 5-ml aliquot add 10 ml of 0·05% arsenazo soln., make up to 100 ml and measure the extinction in a 20-mm cell, with an orange filter, against a known soln. of metallic titanium prepared as described above. The accuracy is within $\pm$0·4%. Beer's law is obeyed for 0·01 to 0·25 mg of Ti in 100 ml. In the presence of oxalates and fluorides the colour does not form.

With tartaric or citric acid in H_2SO_4 the intensity of the colour is slightly reduced, but the sensitivity is the same (0·05 μg of Ti per ml). Alkali and alkaline-earth metals do not interfere. Ferric iron ($> 0·03$ mg) and Cr^{3+} ($< 0·1$ mg) in the test soln. do not interfere.　　　C. D. KOPKIN

2127. Quantitative X-ray spectrographic determination of oxygen in lower titanium oxides. L. M. Kuznetsov, E. S. Makarov and Z. M. Turovtseva (V.I. Vernadskiĭ Inst. Geochem. and Anal. Chem., Acad. Sci. USSR, Moscow). *Zhur. Anal. Khim.*, 1959, **14** (4), 463–465.—The lower titanium oxides containing up to 17% (by wt.) of O ($TiO_{0\cdot 05}$ to $TiO_{0\cdot 60}$, in steps of about $O_{0\cdot 05}$) have been synthesised by the direct reaction of powdered titanium ($\simeq$ 1·5 g) with the calculated amounts of O. The samples were pressed into cylinders under a pressure of 8000 kg per sq. cm and heated at 1000° $\pm$ 20° for 15 hr. to ensure the homogeneous distribution of O. The oxides were finely ground and heated in a vacuum at between 500° and 600° for 30 min. to remove lattice distortions, and the X-ray powder spectrograms obtained. The lattice constants a and c were calculated and a graph of O content *vs.* lattice constant c was drawn; this serves as a calibration curve, by using which O can be determined in lower titanium oxides with an accuracy of $\pm$ 0·1% (by wt.).　　C. D. KOPKIN

2128. Analysis of yttrium metal and yttrium oxide. II. Titanium. J. O. Hibbits, W. F. Davis and M. R. Menke (General Electric Co. Aircraft Nucl. Propulsion Dept., Cincinnati). *U.S. Atomic Energy Comm.*, Rep. APEX-523, Oct., 1959. 15 pp. —A method for the determination of trace amounts of Ti is presented. The method involves the extraction of the titanic thiocyanate complex from 6 M HCl with a soln. of 0·01 M tri-n-octylphosphine oxide in *cyclo*hexane. Of the 31 metallic elements investigated, only Nb, Mo, W and Ta seriously interfered.　　　NUCL. SCI. ABSTR.

2129. Thiodiglycollic acid as a reagent for zirconium. Suseela B. Sant and Bharat R. Sant (Carr Chem. Lab., Mt. Holyoke Coll., Hadley, Mass., U.S.A.). *Anal. Chim. Acta*, 1959, **21** (3), 221–223 (in English).—Zirconium can be pptd. with the reagent from soln. 0·2 to 0·4 N in HCl or HNO_3. The ppt. is ignited and weighed as ZrO_2. Cobalt, Ni, Zn, Mg, Cd, Al, Mn, Pb, Ti, Be and the alkaline-earth metals do not interfere, and the error due to VO^{2+}, $UO_2{}^{2+}$, Fe^{II} and Cr^{III} is negligible if the pptn. is carried out in 0·4 N HCl, but Ce^{IV} must be reduced to Ce^{III} with H_2O_2 before pptn. Sulphate interferes.　　　　　　　W. T. CARTER

2130. Studies in polarographic analysis. XXXIV. Determination of zirconium with Mordant blue 2R. Masayoshi Ishibashi, Taitiro Fujinaga and Kosuke Izutsu (Chem. Dept., Fac. Sci., Kyoto Univ., Sakyo-ku). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (6), 628–631.—In the presence of Zr, the reduction wave of Mordant blue 2R (C.I. Mordant Blue 9) (**I**) at 0 V *vs.* the S.C.E. is shifted to $-0·2$ V at pH 1·5 and the height of the second wave is proportional to the concn. of Zr ($< 10^{-4}$ M). The $E_{\frac{1}{2}}$ value increases linearly with rise of pH and the wave height *vs.* pH diagram gives a max. at pH 1·5. At this pH, the molar ratio of Zr to **I** is 0·5. The presence of $PO_4{}^{3-}$, F^-, As^{III}, Ce^{IV}, Cr^{VI}, Fe^{III}, Mo^{VI}, Sb^{III}, Ti^{IV}, U^{VI} and V^V causes interference. For purification, the Zr is pptd. with M mandelic acid (2·5 ml) (Kumins, *Anal. Chem.*,

1947, **19**, 376) and the ppt. is treated with $2 N$ NaOH (2 ml); the washed $Zr(OH)_4$ is dissolved in 60% $HClO_4$ (0·5 ml), and $2 N$ NaOH (1·0 ml) and 0·003 M **I** (2·0 ml) are added. The mixture is made up to 25 ml, warmed at 75° for 5 min., cooled and submitted to polarography.   K. Saito

2131. Colorimetric determination of zirconium in ores containing phosphates. L. I. Kononenko and N. S. Poluéktov (Inst. of Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1959, **25** (9), 1050–1053.—The sample (0·1 to 0·5 g) of ore is fused with 2 g of $Na_2B_4O_7$ and 3 g of Na_2CO_3 and the matter insoluble in hot water is dissolved in 40 to 50 ml of 4·5 N HCl. The nearly boiling soln. is treated with a few drops of H_2O_2 soln. and 5 ml of 10% ammonium phosphate soln., and set aside in a warm place for 2 hr. The ppt. is collected and washed with 2% NaCl soln. and dissolved in 10 to 15 ml of hot 5% oxalic acid soln. The soln. so obtained is treated with 10% NaOH soln. to give a pink colour with phenolphthalein, then boiled and set aside for 30 min. The ppt. is collected and washed with 2% NaCl soln. and dissolved in 13·5 ml of 5 N HCl. The residual oxalic acid is destroyed with $KMnO_4$, the excess of which is destroyed with hydroxyammonium chloride. To determine the Zr, an aliquot (10 ml) of the soln. (diluted to 50 ml) is mixed with 2·5 ml of 5 N HCl and 5 ml of 0·1% alizarin red soln. and boiled for 3 min. The extinction of the soln., diluted to 50 ml, is measured at 570 mμ. Separation from Nb, but not from Ta, is complete. The method is applicable over the range from 0·01 to 2% of ZrO_2.   G. S. Smith

2132. Spectrographic analysis by evaporation. VIII. Analysis of zirconium. A. N. Zaidel', L. V. Lipis and K. I. Petrov. *Zhur. Anal. Khim.*, 1959, **14** (4), 497–500.—The method of evaporation may be used to determine small amounts of Al, B, Bi, Cd, Cr, Fe, K, Na, Li, Mg, Mn, Ni, Pb, Sb, Si and Sn in zirconium oxide. The use of a vacuum gives best results, except in the determination of Bi, Cd, Pb, Sb and Sn. Reproducible results are attained by using Ga and Co as internal standards in an amount of 1×10^{-3}%, the reproducibilities being (for evaporation in air)—Mn and Cr 8%, B 9%, Fe, Ni, Mg, Si, Bi, Sb, Sn and Pb 10%, Cd 11%, and Al, K, Na and Li 20%. The analytical lines and determinable concn. of these elements are given.   C. D. Kopkin

2133. Determination of zirconium in the presence of niobium and tantalum by the phosphate and 8-hydroxyquinoline methods. A. V. Vinogradov and V. S. Shpinel'. *Zavod. Lab.*, 1959, **25** (9), 1067–1068.—By means of radioactive isotopes it is confirmed that, in the absence of H_2O_2, Nb and Ta are completely pptd. with Zr by the use of $(NH_4)_2HPO_4$. In the presence of H_2O_2 both Ta and Nb are partially co-pptd., the amount of Nb in the ppt. increasing with the amount of Zr present. By the use, first of PO_4^{3-} and then of 8-hydroxyquinoline, nearly complete separation of small amounts (*e.g.*, 2·88 mg) of Zr from Ta and Nb is possible.   G. S. Smith

2134. Application of X-ray fluorescence methods to the analysis of Zircaloy. R. W. Ashley and R. W. Jones (Chem. and Metall. Div., Atomic Energy of Canada Ltd., Chalk River, Ontario, Canada). *Anal. Chem.*, 1959, **31** (10), 1632–1635.—Two alternative X-ray methods have been used for determining Sn, Fe, Cr and Ni in Zircaloy-2. In one, the sample is briquetted with cellulose powder, and, in the other, the bulk of the Zr is removed by extraction with thenoyltrifluoroacetone before the alloying elements are pptd. and briquetted with cellulose powder. The first method is the quicker but less sensitive, and both methods are more rapid than chemical techniques although comparable with them in precision and accuracy. At concn. >200 p.p.m., Hf can also be determined similarly.   K. A. Proctor

2135. Spectrophotometric determination of traces of zirconium and hafnium with Alizarin red S. Yoshio Hoshino (Res. Lab. for Resources Utilisation, Inst. of Tech., Ookayama, Meguro-ku, Tokyo). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (7), 738–743.—The deviation of the calibration curve from Beer's law in the determination of small amounts ($< 20 \mu$g) of Zr and Hf with Alizarin red S (C.I. Mordant Red 3) (**I**) appears to be due to the low rate of reaction. The best conditions are as follows. After the addition of **I** (0·05%, 2 ml) the soln. (50 ml, 0·1 N in HCl) is heated at 100° for a few minutes and the extinction is measured at 500 mμ. The Zr and Hf must not be kept for long in dil. HCl soln. before **I** is added. The interference of Fe^{3+} is avoided by reduction with thioglycollic acid (**II**) (1%, 1 ml per 2 mg of Fe). Zirconium and Hf in H_2SO_4 are co-pptd. with Fe (2 mg), dissolved in 0·1 N HCl and treated with **I** and **II**.   K. Saito

2136. Spectrographic determination of impurities in zirconium and Zircaloy using a chromium internal standard. J. F. Frain, J. R. Ryan and R. M. Jacobs (Westinghouse Electric Corp., Bettis Plant, Pittsburgh, Pa.). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-162-7, June, 1958. 6 pp.—The determination of four impurity elements (Co, Pb, Mo and Ti) in a zirconium oxide matrix is described. The method is applicable to the analysis of metal samples that have been removed from ingots, strips, bars or other shapes and that can easily be converted into the oxide. Synthetic standards containing the appropriate alloying constituents must be used for the analysis of Zircaloys. Metal samples are converted to the oxide by ignition, and a 1-g portion of the oxide is mixed with 100 mg of chromic oxide. The mixture is pressed into the sample electrode and excited in a d.c. arc. The spectra are recorded photographically and measured photometrically; the calculated relative intensity ratios are referred to the appropriate calibration curves.   Nucl. Sci. Abstr.

2137. Spectrochemical determination of impurities in zirconium and Zircaloy using the silver chloride carrier technique. R. F. Farrell, R. M. Jacobs and N. E. Gordon (Westinghouse Electric Corp., Bettis Plant, Pittsburgh, Pa.). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-162-1(Rev. 3), June, 1959. 9 pp.—Before analysis all samples are ignited in a muffle-furnace at 900°. The oxides formed are mixed with a silver chloride carrier, charged into Scribner - Mullin type sample electrodes and exposed with the use of d.c. arc excitation. The spectra are recorded and measured in the usual way. The measured transmittancies for the spectral lines are converted to relative intensities which are referred to calibration curves. The method is applicable to the analysis of metal samples that have been removed from ingots, strips, bars or other shapes and that can be readily converted into the oxide.   Nucl. Sci. Abstr.

2138. Determination of micro amounts of impurities in zirconium by means of basic dyestuffs. A. K. Babko and P. V. Marchenko (Inst. of Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1959, **25** (9), 1047–1050.—*Procedure for Zn*—The sample (0·5 to 2 g) (containing $<10^{-5}$ % of Zn) is dissolved in 5 ml of dil. HF (1:5) with the subsequent addition of a few drops of HNO_3, and the soln. is evaporated to dryness; the residue is dissolved in 10 ml of dil. H_2SO_4 (1:3), 0·5 g of H_3BO_3 is added, the soln. is evaporated to fuming, and the cooled residue is dissolved in 40 ml of water. The soln. in a separating-funnel is treated with 2 ml of 0·6% methylene blue soln. and 10 ml of M NH_4SCN. Flotation of the ppt. is effected by the addition of 4 ml of a mixture of diethyl ether and toluene (1:1) and shaking. The aq. layer is removed and is again subjected to flotation. The ppt. are washed with 1% NH_4SCN soln. and dissolved in 2 ml of dil. H_2SO_4 (1:3). The soln. is treated with 1 ml of 30% H_2O_2 soln. and evaporated to fuming, the treatment with H_2O_2 and evaporation being repeated, if necessary, to give a colourless liquid. The diluted soln. is adjusted with aq. NH_3 to a pH of $\simeq 6$ and the Zn is determined by means of dithizone. *Procedure for Cd*—The sample (1 to 2 g) is dissolved as described for Zn, and the residue after evaporation of the acids is dissolved in 10 ml of dil. HCl (1:5). The soln. in a separating-funnel is mixed with 35 ml of water, 10 ml of M KI and 5 ml of 0·6% methyl violet soln. and the ppt. is subjected to flotation with 4 ml of toluene. The ppt. is washed with 1% KI soln., and dissolved in H_2SO_4 in the presence of H_2O_2. The Cd is determined by means of dithizone in alkaline soln. There is interference from Cu, Bi and Pb, but not Zn. *Procedure for Mo and Bi*—The sample (2 g) is dissolved in 10 ml of dil. H_2SO_4 (1:4) with the dropwise addition of 8 ml of dil. HF (1:1). The soln. is evaporated to incipient crystallisation, the residue is dissolved in 10 ml of water and the soln. is treated with dil. aq. NH_3 (1:1) in the presence of two drops of 0·3% methyl violet soln. to give a blue - green colour (pH $\simeq 1$). The soln. in a separating-funnel is treated with 10 ml of M NH_4SCN and 0·3% methyl violet soln. (2 × 3 ml) and flotation is carried out with 5 ml of toluene. The ppt. is dissolved and the dyestuff oxidised as described above. Aliquots of the aq. soln. are used for determining Mo by the colorimetric thiocyanate method, in the presence of ascorbic acid as reducing agent, and Bi by the iodide method in the presence of thiourea.

G. S. SMITH

2139. Spectrographic determination of sodium, lithium, barium and strontium in zirconium and Zircaloy using the silver chloride carrier technique. R. F. Farrell and G. J. Harter (Westinghouse Electric Corp. Bettis Plant, Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-162–14, Apr., 1959. 7 pp.—The elements determined and the concn. ranges in p.p.m. are—Na, 1 to 100; Li, 2 to 100; Ba, 1 to 100; and Sr, 1 to 100. The method is applicable to the analysis of metal samples that have been removed from ingots, strips, bars or other shapes, and that can be readily converted into the oxide. Synthetic standards containing the appropriate alloying constituents must be used for the analysis of Zircaloys. By varying the exposure and excitation parameters, K may be determined by the same general method.

NUCL. SCI. ABSTR.

2140. Spectrochemical determination of aluminium and copper in zirconium and Zircaloy using d.c. arc excitation and a barium fluoride flux. R. M. Jacobs, R. F. Farrell and T. F. Anater (Westinghouse Electric Corp., Bettis Plant, Pittsburgh, Pa.). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA-(GLA)-162–5 (Rev. 3), July, 1959. 5 pp.—This method is used for the spectrographic analysis of zirconium and Zircaloy oxides. The concn. ranges covered are, for Al, 10 to 300 p.p.m. and, for Cu, 10 to 200 p.p.m. All samples are ignited at 900° and the oxides so formed are mixed with a barium fluoride - graphite buffer and are then exposed by using d.c. arc excitation. The spectra are recorded and measured by standard methods. The measured line transmittancies are converted to relative intensities and referred to calibration curves.

NUCL. SCI. ABSTR.

2141. Determination of carbon in zirconium and zirconium-base alloys. K. A. Morrow and E. F. Tretow (Westinghouse Electric Corp., Bettis Plant, Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-181 (Rev. 2), Apr., 1959. 5 pp. —The sample is burnt in a stream of O by using induction heating, and the CO_2 in the evolved gases is absorbed in dil. NaOH soln. The amount of C present in the sample is determined by measuring the change in conductance of the absorbing soln. and referring this change to a standard curve. The method covers the determination of C in zirconium and Zircaloy in a concn. range from 10 to 800 p.p.m. on a 0·5-g sample basis. Satisfactory results may be obtained on sample portions as small as 0·1 g, thus extending the upper limit to 4000 p.p.m. of C. Sample segregation must be taken into consideration whenever smaller samples are used.

NUCL. SCI. ABSTR.

2142. Photometric determination of silicon in zirconium. S. V. Elinson and L. I. Pobedina. *Zavod. Lab.*, 1959, **25** (8), 909–911.—The sample (0·2 g) is fused with $K_2S_2O_7$, the melt is dissolved in 40 to 50 ml of water containing 1·5 ml of conc. H_2SO_4, and an aliquot ($\not> 10$ ml) of the diluted soln. (100 ml), containing between 2 and 40 μg of Si, is diluted to between 30 and 35 ml and treated with 3 ml of 5% $(NH_4)_2MoO_4$ soln. After 10 min., 4 ml of 8 N H_2SO_4 and 2 ml of 1% ascorbic acid soln. are added, the soln. is diluted to 50 ml and the extinction is measured after 30 min., with a yellow filter. With contents $>0·3$%, any Si present in the matter not dissolved during the inital attack with acid must be recovered.

G. S. SMITH

2143. Determination of tin in zirconium-base alloys. T. M. Reinhold (Westinghouse Electric Corp., Bettis Plant, Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-371(Rev. 2), Aug., 1959. 4 pp.—Tin is reduced to the stannous state with aluminium in HCl - HF medium. Stannous tin is titrated with standard iodate - iodide soln., with starch as indicator. This method covers the determination of tin in Zircaloy-2 (1·25 to 1·65%) and Zircaloy-3 (0·20 to 0·30%).

NUCL. SCI. ABSTR.

2144. Determination of titanium as an impurity in zirconium and Zircaloy. D. V. Reed (Westinghouse Electric Corp., Bettis Plant, Pittsburgh, Pa.). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA-(GLA)-157(Rev. 2), May, 1959. 5 pp.—In conc. H_2SO_4, 5-sulphosalicylic acid forms a yellow

complex with Ti which can be measured spectro-photometrically at 450 mμ. The colour is stable for $<$24 hr. Positive interferences from Fe and V are negligible provided that the material contains $<0.2\%$ of Fe and <100 p.p.m. of V. Fluoride from HF dissolution is reduced to a non-interfering level by fuming the sample with H_2SO_4. The interference of coloured ions is corrected for by the use of a sample soln. blank. The method covers the determination of 10 to 100 p.p.m. of Ti on a 1-g sample.   Nucl. Sci. Abstr.

2145. **Determination of oxygen as an impurity in zirconium and Zircaloy.** P. Elbling and G. W. Goward (Westinghouse Electric Corp., Bettis Plant, Pittsburgh, Pa.). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-592 (Rev. 1), July, 1959. 12 pp. —The metal sample is caused to react with carbon in molten platinum in a graphite crucible. The carbon reacts with the oxides in the sample to form CO, which is swept out of the reaction tube with a stream of purified argon. Iodine pentoxide oxidises the CO to CO_2, which is absorbed by a dil. NaOH soln. The amount of oxygen present in the sample is determined by measuring the change in conductance of the absorbing soln. and referring this change to a standard curve prepared by the analysis of known amounts of stoicheiometric metal oxides such as those of Ag, U, Nb and Zr. The method covers the range from 50 p.p.m. (1-g sample) to 7000 p.p.m. (0.1-g sample). Nucl. Sci. Abstr.

2146. **Spectrographic determination of tin, iron, chromium and nickel in Zircaloy-2 using high-voltage-spark point-to-plane excitation.** R. M. Jacobs and N. E. Gordon (Westinghouse Electric Corp., Bettis Plant, Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-162-12, Oct., 1958. 6 pp.—The method is applicable to the analysis of any metal specimen that has been removed from ingots, strips or bars, and that can be prepared for analysis by machining or grinding to give a smooth flat surface. The sample is placed on a Petrey stand where excitation is achieved by using the appropriate high-voltage-spark parameters. The emitted spectral radiation is dispersed in a large Littrow prism spectrograph and the spectra are recorded photographically. Densitometric measurements and calculations are made according to standard spectrochemical practices.   Nucl. Sci. Abstr.

2147. **Determination of uranium as an impurity in zirconium and Zircaloy.** D. L. Smith, H. R. Wilson and G. W. Goward (Westinghouse Electric Corp., Bettis Plant, Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-431 (Rev. 2), Aug., 1959. 5 pp.—Samples are dissolved in HNO_3 and HF. The soln. are satd. with $Al(NO_3)_3$, and the U is extracted with diethyl ether. The U is stripped from the ether layer into water, and an aliquot of this soln. is fused with a mixture of sodium and lithium fluorides. The fluorescence produced by the U is measured with a photo-electric fluorimeter. This method covers the determination of U in zirconium and Zircaloy in the range 0.3 to 5 p.p.m. on a 1-g sample basis. These limits can be varied by changing the size of the aliquot used for the final U determination. (See also *Anal. Abstr.*, 1959, 6, 909.)   Nucl. Sci. Abstr.

2148. **Gravimetric determination of hafnium and zirconium in hafnium materials.** T. M. Reinhold (Westinghouse Electric Corp., Bettis Plant, Pitts-burgh, Pa.). *U.S. Atomic Energy Comm.*, Rep. WAPD-M(GLA)-718, April, 1959. 4 pp.—Hafnium and Zr are pptd. by mandelic acid in HCl soln. The mandelates are ignited and weighed as oxides. This method covers the determination of the sum of the two metals in raw and purified hafnium oxides, hafnium sponge and refined metal. The method is accurate to $\simeq \pm0.3\%$ (relative).
        Nucl. Sci. Abstr.

2149. **Determination of the primary impurities in hafnium by emission-spectrographic analysis.** J. F. Frain, J. R. Ryan and R. M. Jacobs (Westinghouse Electric Corp., Bettis Plant, Pittsburgh, Pa.). *U.S. Atomic Energy Comm.*, Rep. WAPD-M(GLA)-701-1 (Rev. 2), Aug., 1959. 11 pp.—A procedure is described for the determination of 14 impurity elements in hafnium metal that has been converted into the oxide by ignition. A portion (0.5 g) of the sample is mixed with 75 mg of a buffer. This mixture is pressed into the sample electrode and excited in a d.c. arc. The spectra are recorded photographically and measured photometrically, and the calculated intensity ratios are referred to the appropriate calibration curves.
        Nucl. Sci. Abstr.

2150. **Determination of the secondary impurities in hafnium by emission-spectrographic analysis.** J. F. Frain and J. R. Ryan (Westinghouse Electric Corp., Bettis Plant, Pittsburgh, Pa.). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA-M(GLA)-701-2, April, 1959. 7 pp.—A procedure for the determination of 12 impurity elements in a hafnium oxide matrix is described. The procedure is applicable to the analysis of hafnium that has been converted into the oxide. The elements determined and the concn. ranges are tabulated.
        Nucl. Sci. Abstr.

2151. **Use of o-cresotic acid as a reagent for the estimation of thorium and zirconium.** T. N. Srivastava, S. P. Agarwal and R. C. Aggarwal (Chem. Dept., Lucknow, India). *Z. anal. Chem.*, 1959, **169** (4), 254–257 (in English).—Both Th (>10 mg) and Zr (>10 mg) are quant. pptd. at pH 3.7 to 5.2 by hot aq. o-cresotic acid (6-hydroxy-o-toluic acid) (**I**) or by its sodium salt. At lower pH values pptn. is incomplete. While Th forms a well-defined salt containing equimolar amounts of Th and **I**, suitable for direct weighing after drying at 105° to 110°, the zirconium ppt. is a basic salt of variable composition and suitable only for ignition to ZrO_2. In the determination of Th and Zr, no interference is caused by Be, Ca, Ba, Mg, Zn, Cd, Hg^{2+}, Pb, Co, Cu^{2+}, Mn, Ni, Bi, Li, Ce^{3+} and Al; that due to UO_2^{2+}, Cr^{3+} and Fe^{3+} is avoided by double pptn., that due to Ce^{4+} by reduction to Ce^{3+} with H_2SO_3, and that due to Ti^{4+} by complexing with H_2O_2 and double pptn. At pH >4.27, **I** quant. reduces Au^{III} to metallic gold and can thus be used for determining Au.   J. P. Stern

2152. **Complexometric and photometric determination of thorium in minerals and ores.** F. V. Zaĭkovskiĭ. *Zhur. Anal. Khim.*, 1959, **14** (4), 440–444.—To the sample (5 to 50 mg) in a 10-ml porcelain crucible containing $\simeq 1$ g of Na_2O_2 and 4 to 5 mg of Fe_2O_3 add 1 to 2 g of Na_2O_2 and fuse at 550° to a homogeneous melt (2 to 3 min.). Cool, treat the melt with water (50 to 80 ml) at 100° for 10 min.; after 25 min. filter, wash the ppt. with 0.5% NaOH soln. ($\times 2$ or 3), dissolve it on the filter with hot HNO_3 (1:2) (9 ml) and hot water (10 ml), wash the filter with hot water (5 to 6 ml), add 1 drop

of H_2O_2 to the soln., cool to between 1° and 5° and precipitate Th by adding 60% tartaric acid soln. (5 ml), 3% $Hg(NO_3)_2$ soln. (0·5 ml), 6% KIO_3 soln. (10 ml) and water to 50 ml. Set aside in a cold place for 45 min., filter off the ppt. with paper-pulp, and wash it with KIO_3 soln. (500 ml containing 6 g of KIO_3, 3 ml of H_2O_2 and 30 ml of conc. HNO_3) (20 to 25 ml). Dissolve the ppt. on the filter with 20% (v/v) HCl (10 to 15 ml), wash the filter with hot water (10 to 15 ml) ($\times$ 2 or 3), collect the filtrate in the beaker in which pptn. of Th took place, and evaporate it to 1 ml. With large amounts of Th, Zr and Ti, use oxalate pptn. (see below); with small amounts of these elements, add conc. HCl (5 or 6 ml) and 40% (w/v) formaldehyde soln. (5 or 6 drops) and evaporate to 0·5 ml, repeating this operation to remove the iodine completely. Add water (6 to 8 ml), aq. NH_3 to pH 2 or 3 (Congo red), 1% ascorbic acid soln. till the soln. is colourless and a further 0·2 ml, filter into a 25-ml flask, make up to volume with 0·09 N HCl, and determine Th photometrically with arsenazo and tartaric acid as described previously (*cf.* Zaĭkovskiĭ and Gerkhardt, *Anal. Abstr.*, 1959, **6**, 1260). *Oxalate pptn.*—To the residue obtained after the evaporation with HCl (see above) add water (30 or 40 ml) and 25% $CaCl_2$ soln. (0·5 ml), and satd. Na acetate soln. dropwise to a pH of 5 (Congo red paper). Dilute to 50 ml, add dioxalylacetone soln. (7·5 ml) and proceed as described previously (*loc. cit.*). Determine Th in the resulting soln. photometrically as before, or complexometrically as follows. To 10 to 15 ml of the test soln. add 30% KSCN or NH_4SCN soln. (1 ml); the resulting colour due to the presence of Fe is used as indicator. Prepare a compensating soln. containing the same volume of water as the test soln., 2 drops of HCl and 1 ml of the thiocyanate soln. To this soln. add 3 drops of $FeCl_3$ soln. (10 mg of Fe in 100 ml), and adjust the colours of the test and compensating soln. until they are identical by adding 0·001% ascorbic acid soln. or $FeCl_3$. Heat the compensating soln. to 60° and slowly titrate with 0·002 M or 0·005 M EDTA (disodium salt) till colourless; this is the blank. Titrate the test soln. similarly till colourless. The complexometric method has been tested on various minerals (monazite and others) with an error (relative) of ±6%. The photometric method has been used for minerals containing 0·005 to 4% of Th and 60 to 70% of Zr and Ti, with errors of ±11% (relative).

C. D. Kopkin

2153. Extraction - photometric determination of thorium in natural materials. L. I. Gerkhardt. *Zhur. Anal. Khim.*, 1959, **14** (4), 434–439.—At pH 2·5 to 4·5, Th forms with phenylcinchoninic acid (**I**) or its ammonium salt the compound $Th(C_6H_5C_9H_5NCOO)_4.C_6H_5C_9H_5NCOOH$, which is insol. in water but sol. in alcohols, ethers and ketones; the soln. in butanol (**II**) may be used to determine Th with arsenazo (**III**). Addition of ascorbic acid (**IV**) to the aq. soln. causes the formation of the complexes of Ti, Zr and U with **IV**, which are not extracted into **II**. **IV** reduces Fe^{III} to Fe^{II}, which does not react with **I**. Bivalent ions do not react with **I** and are not extracted with **II**; in the presence of **IV**, tervalent ions, including those of rare-earth elements, V, Mo and W, do not react with **I** and are not extracted with **II**. *Procedure*—Fuse the ore or mineral (5 to 50 mg) with Na_2O_2 (1·5 to 2 g) at 550°. Cool the homogeneous melt, dissolve it in water (30 ml), heat the soln. for 10 to 15 min., filter, wash the ppt. with cold water

($\times$ 2 or 3) and dissolve it on the filter with hot 5% (v/v) HCl (5 to 10 ml). Adjust the soln. with aq. NH_3 to pH 2·5 or 3·0 (Congo red), and make up to 25 ml with 0·09 N HCl. Transfer 2 to 10 ml of the resulting soln. to a 20 to 50-ml separating-funnel, add **IV** (1 to 2 mg) and M ammonium acetate (2 ml) and make up to 10 ml with 0·09 N HCl. Mix well, add 2% **I** (ammonium salt) soln. in **II** (5 ml) and **II** (5 ml) and extract the Th for 20 to 40 sec. Transfer the organic phase to a 25-ml flask, wash the funnel with acetone (5 to 10 ml), and to the combined soln. add 0·05% **III** soln. (3 ml), **IV** (1 to 5 mg) and 10% tartaric acid soln. (0·5 ml), and make up to volume with 0·09 N HCl. After 1 to 2 hr. measure the extinction in a 30-mm cell at 670 mμ (yellow - green filter), and refer the results to a calibration curve prepared from soln. containing 0, 5, 10, 15 and 70 μg of Th. From 0·005% of Th may be determined, with an error (relative) of 5 to 20%.

C. D. Kopkin

2154. Absorptiometric determination of thorium in miscellaneous plant products including ores, chemical and gravity concentrates, uranium trioxide and various plant streams. R. C. Sackville (Eldorado, Saskatchewan). Rep. AM-117, Eldorado Mining and Refining Ltd., Apr., 1956. 9 pp.—This method covers the various initial treatments involved for each type of sample, and the common absorptiometric finish on the final soln. obtained. Ores, gravity concentrates and mine leach ppt. are fused with Na_2O_2 and separations carried out involving aq. NH_3 and KIO_3. Uranium trioxide and diuranate ppt. are simply dissolved in acid and diluted to vol. Aqueous samples such as slurries, raffinates and scrub liquors can generally be diluted and treated directly, provided that the content of rare-earth elements is low compared with that of Th. Solvent samples are stripped twice with water and diluted as required. Aliquots from these prepared soln. are then taken and the Th is pptd. as the fluoride on a lanthanum carrier. A large dilution factor and the fluoride pptn. serve to reduce or eliminate most interfering cations. Fuming to dryness with $HClO_4$ removes the remaining interfering anions. The residue is dissolved in HCl, hydroxyammonium chloride is added, and an orange - red colour is developed by the addition of thoron reagent. The extinction of this Th - thoron complex is measured spectrophotometrically at 545 mμ.   Nucl. Sci. Abstr.

2155. Determination of thorium in plutonium - thorium alloys. K. S. Bergstresser and M. E. Smith (Los Alamos Sci. Lab., N. Mex.). *U.S. Atomic Energy Comm.*, Rep. LA-1839 (Del.), Sept., 1954. Decl. with deletions Oct., 1956. 25 pp.—A spectrophotometric method for the determination of Th was modified for the purpose of analysing plutonium alloys which contained 0·01 to 0·8% of Th. Extinction values were measured at 545 mμ for soln. containing Th and thoron, the organic reagent added to form a coloured complex with Th in the presence of Pu^{III}. With samples of Pu - Th soln., equivalent to dissolved alloys containing 0·2 to 0·8% of Th, the mean recovery was 99·6 ± 1·5%. For similar samples containing 0·01 to 0·2% of Th, the mean recovery was 101·0 ± 3·4%, after the Th had been separated from the Pu by pptn. of ThF_4 with LaF_3 as carrier.   Nucl. Sci. Abstr.

2156. Ion-exchange method for the separation of radium-228, actinium-228, lead-212 and bismuth-212 from thorium nitrate. Ying-Mao Chen

and Chiu-Ming Wong (Dept. of Chem., Nat. Taiwan Univ., China). *J. Chinese Chem. Soc., Ser. II*, 1959, **6** (1), 55–67 (in English).—After complexing with tartaric acid, citric acid or EDTA (the first giving the greatest recovery) the mixture of isotopes is passed through a cation-exchange column (Dowex 50, Na+ form). The decay products are retained and Th passes through; [228]Ac is then separated by eluting the other three isotopes with $2N$ HCl and is itself recovered by eluting with $4N$ HCl. The $2N$ HCl eluate is passed through an anion-exchange column (Amberlite IRA-400, Cl⁻ form), when [212]Pb and [212]Bi are retained and [228]Ra, passes through. By treating this column with $8N$ HCl, [212]Pb is eluted and [212]Bi is retained, to be removed with $2N$ H_2SO_4.

G. S. ROBERTS

2157. The determination of nitrogen in metals by isotope dilution. H. G. Staley and H. J. Svec (Inst. for Atomic Research, Iowa State College, Ames, U.S.A.). *Anal. Chim. Acta*, 1959, **21** (3), 289–295 (in English).—After the addition of ammonium sulphate having a ten-fold enrichment of ^{15}N, the metal sample is dissolved in acid. The nitrogen from the metal and the tracer is then recovered as NH_3 by distillation from alkaline soln. and oxidised to gaseous nitrogen with alkaline hypobromite. The amount of ^{15}N in the nitrogen is then determined in a mass spectrometer, and the nitrogen content of the metal is calculated from the change in concn. of ^{15}N which has taken place. Air contamination can be detected by monitoring the [40]A⁺ peak and corrected for if necessary. Results are quoted for the analysis of a wide variety of metals and alloys containing from 2 p.p.m. to 6% of N. The average precision is approx. $\pm5\%$.   W. T. CARTER

2158. Determination of nitrogen in nitrocellulose and nitric acid. II. Other gases in the nitric oxide produced by the Lunge method. Tetsuro Murakami (Kogakuin, Tsunohazu, Shinjuku-ku, Tokyo). *Japan Analyst*, 1958, **7** (11), 681–684.—Most of the NO in the gas in the Lunge nitrometer was removed with $FeSO_4$ (saturated soln. in N H_2SO_4) and the residual gas was examined mass-spectrometrically. This was found to consist mainly of N, but it appears that this is derived from the $FeSO_4$ soln., since the ratio of N to A is $\simeq50:1$. This method is therefore unsuitable for the analysis of impurities in the gas.
III. Composition of the gas evolved by the Lunge method. Tetsuro Murakami. *Ibid.*, 1958, **7** (11), 685–690.—By cooling the gas in the nitrometer with liquid air, CO_2, N_2O and a part of the NO are collected and are determined mass-spectrometrically with peaks m/e 22, 44 and 30, respectively. Nitrogen, A, NO and CO are not condensed and are determined with the peaks m/e 28, 40, 30 and 12, respectively. The amount of N_2O increases with the concn. of the H_2SO_4 used for the decomposition of the sample (nitrocellulose or KNO_3) (*e.g.*, 0·22% for 94·5% H_2SO_4 and 0·39% for 98% H_2SO_4). The amount of CO and CO_2 also increases with the concn. of H_2SO_4 in the analysis of nitrocellulose.
IV. The correction for practical analysis. Tetsuro Murakami. *Ibid.*, 1958, **7** (12), 761–766.—The effect of the formation of N_2O ($\simeq0\cdot2\%$ by the use of 94·5% H_2SO_4) is compensated by the residual film of H_2SO_4 on the gas burette. The effects of the solubility of NO in H_2SO_4 and of the formation of

CO and CO_2 increase with increase in concn. of H_2SO_4, but remain almost unchanged with change in temp. An empirical formula is proposed—V (corrected vol.) = V_0 (measured vol. of NO) × 0·998 (correction for CO and CO_2) + V_s (effect of the solubility, 0·64 ml when 18 ml of 94·5% H_2SO_4 is used).
V. Volumetric determination of nitric acid and nitrate by reduction with stannous chloride. Tetsuro Murakami. *Ibid.*, 1958, **7** (12), 766–771.—By being boiled with $0\cdot2N$ $SnCl_2$ (50 ml) in 6 to $8N$ HCl for 30 min. in a current of N, NO_3^- (1·5 to 20 mg of N) are reduced to hydroxylamine and the excess of Sn^{2+} can be titrated with $FeCl_3$ soln. at $>60°$. Dissolved O does not interfere; neither NO nor NO_2 is formed during the reduction. This method is applicable to the analysis of HNO_3 and nitrates, but not to that of nitrocellulose or a mixture of H_2SO_4 and HNO_3 containing NO_2. The time taken for analysis is $\simeq30$ min.   K. SAITO

2159. The gas-chromatographic separation of nitrogen trifluoride from carbon tetrafluoride. E. Nachbaur and A. Engelbrecht (Inst. anorg. und anal. Chem., Univ., Innsbruck, Austria). *J. Chromatography*, 1959, **2** (5), 562–564 (in German).— Complete separation is achieved with hydrogen as carrier gas at a flow rate of 10 ml per min. on a column (12 metres × 4 mm) of moist silica gel at 0°. Partial separation may be achieved on activated carbon (3 metres × 4 mm) at $-21°$ with hydrogen at a flow rate of 45 ml per min.

A. R. ROGERS

2160. Detection of phosphates on chromatograms. H. Rosenberg (Dept. of Biochem., John Curtin Sch. of Med. Res., Aust. Nat. Univ., Canberra). *J. Chromatography*, 1959, **2** (5), 487–489 (in English). —Dilute a 0·5% soln. of V_2O_5 in 0·5 N HCl (20 ml) to 100 ml with acetone, shake with finely powdered zinc (250 mg) in a stoppered flask until the soln. is quite clear and brown, and use the supernatant liquid; this reducing reagent must be prepared immediately before use. Dip the dried paper chromatogram into a mixture of 5% ammonium molybdate soln. - 5 N HCl - acetone (3 : 7 : 90), allow to dry, dip into the reducing reagent and again allow to dry. After $\simeq2$ min. phosphates appear as blue spots on a background which remains white for <24 hr. The test will detect 0·02 μg of P per sq. cm of paper.   A. R. ROGERS

2161. Colorimetric determination of phosphoric acid by the molybdenum blue method with phenylthiosemicarbazide as reducing agent. Sumio Komatsu and Yasuko Iwakuma (Fac. of Lit. and Sci., Univ., Toyama). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (8), 881–883.—In acetic acid (<2 N), molybdophosphoric acid is reduced by phenylthiosemicarbazide (**I**) to molybdenum blue but molybdic acid is not. In the presence of 3 ml of 2·5% $(NH_4)_2MoO_4$ soln. and 30 ml of **I** soln. (1·5 mg per 2 ml of $2N$ acetic acid), the blue soln. is stable for 30 min. and Beer's law is obeyed for 1 to 12 μg of P per ml at 750 mμ and 0·1 to 5 μg per ml at 372 mμ. The error is $<2\%$. The molybdenum blue is extracted with diethyl ether - isoamyl alcohol (1 : 1) (10 ml) from $2N$ H_2SO_4 soln. (50 ml) containing the stated amounts of Mo and **I**; the extinction coeff. of the org. layer is $\simeq10$ times as great as that of the aq. layer.

K. SAITO

2162. New chemi-chromatographic method for determination of phosphate. R. Antoszewski and J. S. Knypl (Inst. Plant Physiology, Univ. Łódź, Poland). *Z. anal. Chem.*, 1959, **169** (4), 269–271 (in German).—Addition of $PO_4{}^{3-}$ to paper impregnated with quinoline molybdate produces a yellow spot, the area of which is proportional to the quantity of $PO_4{}^{3-}$; 1·25% H_2SO_4 is used to wash the spot. The accuracy is improved by contact u.v. photography of the spot through transparent graph paper, and by the use of const. temp.; for $>0·5 \mu g$ of P it is within $\pm 4\%$. J. P. STERN

2163. Complexometric determination of phosphate in conjunction with the Schöniger combustion procedure. R. Bennewitz and I. Tänzer (Anal. Lab., VEB Fettchemie, Karl-Marx-Stadt, Germany). *Mikrochim. Acta*, 1959, (6), 835–840 (in German).— On the basis of a method by Zavarov *et al.* (*Anal. Abstr.*, 1956, **3**, 3605) a simplified modification was made of the indirect complexometric determination of $PO_4{}^{3-}$ which avoids the tedious isolation of the $PO_4{}^{3-}$ by pptn. as $MgNH_4PO_4$ with excess of $MgCl_2$. The excess of $MgCl_2$ can be titrated accurately in 50% ethanol soln. with EDTA, with Eriochrome black T as indicator, since the $MgNH_4PO_4$ is practically insol. in this medium. The method is intended for use in conjuction with the Schöniger combustion procedure (*Mikrochim. Acta*, 1955, 123; 1956, 869). D. F. PHILLIPS

2164. Assay of reagent-grade "metaphosphoric acid". H. A. Flaschka and W. E. Wolfram (Georgia Inst. of Tech., Atlanta, U.S.A.). *Chemist Analyst*, 1959, **48** (3), 65–66.—A soln. of the sample is titrated with 0·1 N NaOH, with bromocresol green as indicator, the end-point being located with the aid of a comparison soln. The sum of HPO_3 and H_3PO_4 is calculated (as HPO_3) from this titre. A second portion of the soln. is passed through a strongly acidic cation-exchange resin, which is then washed with water and the combined percolate and washings, free from Na^+, are titrated as described above. From the difference of the two titrations the $NaPO_3$ content is calculated. G. S. ROBERTS

2165. Studies in qualitative inorganic analysis. XIII. Reduction of arsenic(V) with ammonium iodide and subsequent precipitation of arsenic(III) sulphide. W. M. Dowson (Tech. Coll., Coventry, England). *Mikrochim. Acta*, 1959, (6), 841–846 (in English).—Conditions have been determined for the reduction of As^V with ammonium iodide at low acidity, followed by the pptn. of arsenous sulphide by H_2S on the semi-micro scale. Under such conditions the separation of Cu, Sn^{IV} and Hg as sulphides is not prevented and S^{2-} are not oxidised to $SO_4{}^{2-}$. D. F. PHILLIPS

2166. Ultra-violet spectrophotometric determination of bismuth with hydrobromic acid. Yuroku Yamamoto (Chem. Dept., Fac. of Sci., Kyoto Univ., Sakyo-ku, Kyoto). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (8), 875–878.— By the use of the peak at 265 $m\mu$, the sensitivity of the determination of Bi with HBr is much increased. The peak wavelength is independent of the concn. of HBr ($>2·6 N$). The extinction remains unchanged for 1 hr. and decreases with rise of temp. The working curve is linear for <12 p.p.m. of Bi in 4·3 N HBr. The results are high in the presence of >10 p.p.m. of Fe^{III}, Pb, Sn^{IV} and Sb^V. K. SAITO

2167. Back-titration with mercuric nitrate in alkaline medium. Determination of small amounts of bismuth and analysis of its binary mixtures with some other metals. H. Khalifa and A. Soliman (Nat. Res. Centre, Dokki, Egypt). *Z. anal. Chem.*, 1959, **169** (2), 109–115 (in English).—Bismuth (0·1 to 25 mg) is determined by addition of excess of EDTA (disodium salt) and back-titration at pH 8·5 to 10 with $Hg(NO_3)_2$ soln., with an error $< \pm 0·1\%$. Binary mixtures with Mg, Sr or Ba are analysed by back-titration at pH 8·5 for the determination of Bi, and then at pH 10·2 to 10·75 for Bi plus the other metal. Binary mixtures with Ca, Cd, Zn, Cu, Pb, Ni, Al, Mn or La are analysed by titration with 0·02 M EDTA (disodium salt) at pH 2·5 in the presence of 0·5% KI soln. (3 drops plus a further 7 to 8 drops just before the end-point) for the determination of Bi alone, followed by addition of excess of EDTA and back-titration with $Hg(NO_3)_2$ soln. at pH 8·5 to 9·5 for determination of both components. J. P. STERN

2168. Photometric determination of vanadium with hydrogen peroxide. H. Hartkamp (Inst. f. Spektrochem. u. angew. Spektroskopie, Dortmund-Aplerbeck, Germany). *Z. anal. Chem.*, 1959, **169** (5), 339–342 (in German).—The absorption maximum of the colour obtained with V^V and H_2O_2 in acid soln. shifts according to the concn. of H_2O_2 and acid. Measurement at 405 $m\mu$, however, gives extinctions which are independent of the concn. of H_2SO_4 between 0·05 M and M, and of H_2O_2 between 0·01% and 1·5%. H. M.

2169. Colorimetric determination of vanadium in niobium - vanadium alloys. O. J. Articolo (Knolls Atomic Power Lab., Schenectady, N.Y.). *U.S. Atomic Energy Comm.*, Rep. KAPL-M-OJA-1, June, 1959. 23 pp.—A procedure is described for the determination of V ($\leqslant 0·2\%$) with an accuracy within 3%. The method was applied to the analysis of niobium alloys in which the nominal V content varied between 0·3 and 4·6%. The sample is dissolved in a mixture of HNO_3 and HF and then evaporated to fumes with H_2SO_4. The Nb is hydrolysed with H_2SO_3 and separated from the V by filtration; H_2O_2 is added to the filtrate to form a reddish-brown complex with the V. The extinction of the soln. is measured at 450 $m\mu$ with a model B Beckman spectrophotometer.　NUCL. SCI. ABSTR.

2170. An ultra-violet spectrophotometric method for the determination of small amounts of vanadium in ores and steel. R. J. Warren, J. F. Hazel and W. M. McNabb (Dept. of Chem., Univ. of Pennsylvania, Philadelphia, U.S.A.). *Anal. Chim. Acta*, 1959, **21** (3), 224–226 (in English).—Vanadium is determined by the absorption of the orthovanadate ion at 270 $m\mu$ in N NaOH. Interference from chromate is eliminated by an ion-exchange separation. *Procedure for ores*—Fuse the sample (1 g) with Na_2CO_3 (5 g), extract with hot water, add methanol (5 ml), boil for 5 min. and filter. Adjust the soln. to pH 2 with HCl and dilute to 100 ml. Pass a 20-ml aliquot through a column of Amberlite IRA-400 (chloride form) and wash through with water. Add sufficient NaOH to the eluate to give a N soln. and dilute to 100 ml. Measure the extinction at 270 $m\mu$ and read off the vanadium concn. from a calibration curve. *Procedure for steel*—Dissolve the sample (1 g) in 6 N HCl (10 ml). Evaporate almost to dryness, add 2 drops of conc. HNO_3 and pour into hot 2·5 N NaOH (40 ml).

Dilute to 100 ml, allow the ppt. to settle and take a 20-ml aliquot. Adjust to pH 2 with HCl and complete the determination as for ores. Results are quoted for a series of ores of known vanadium content in the range 0·05 to 0·59%.

W. T. CARTER

2171. Direct estimation of niobium by N-benzoyl-N-phenylhydroxylamine. A. K. Majumdar and A. K. Mukherjee (Dept. of Inorg. and Anal. Chem., Jadavpur Univ., Calcutta). *Anal. Chim. Acta*, 1959, **21** (3), 245–247 (in English).—The complex obtained by the pptn. of niobium with the reagent (*cf. Anal. Abstr.*, 1958, **5**, 1821) has the composition $NbO(C_{13}H_{10}O_2N)_3$ and is stable up to 229°. After being dried at 110° it can be used as a form for weighing Nb. W. T. CARTER

2172. Determination of niobium in the presence of large amounts of titanium. Yu. I. Bȳkovskaya. *Trudȳ Komiss. Anal. Khim.*, 1958, **9** (12), 329–332; *Ref. Zhur., Khim.*, 1959, (12), Abstr. No. 27,047.— Methods for the determination of Nb in the presence of large amounts of Ti based on differential hydrolysis (Oshman, *Zavod. Lab.*, 1946, **2**, 154) and on the pptn. of Nb by tannin in the presence of ascorbic acid (Ponomarev and Sheskol'skaya, *Anal. Abstr.*, 1958, **5**, 466) were tested with the radio-isotope ⁹⁵Nb. In the first method, with a sample containing 0·023 g of Nb (Nb to Ti = 1:3), 10·22 to 11·20% or 0·00215 to 0·00257 g of Nb remained in the soln. after hydrolysis. In the second method, with a sample containing 0·0027 g of Nb (Nb to Ti = 1:44), 5·1 to 6·5% or 0·0019 to 0·0024 g of Nb remained unpptd. in the reaction mixture. K. R. C.

2173. Spectrographic determination of impurities in niobium. J. A. Featheringham, C. F. Lentz and R. M. Jacobs (Westinghouse Electric Corp., Bettis Plant, Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-631-1, Aug., 1958. 8pp.— The 22 impurities and concn. ranges are listed. The method is applicable to the analysis of metal samples that can be converted into the oxide by ignition. NUCL. SCI. ABSTR.

2174. Spectrographic determination of boron and cadmium in niobium. J. A. Featheringham, C. F. Lentz and R. M. Jacobs (Westinghouse Electric Corp., Bettis Plant, Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-631-3, Aug., 1958. 3 pp.—The method is applicable to the analysis of metal samples that can be converted into the oxide by ignition. A summary of the procedure is given. NUCL. SCI. ABSTR.

2175. Determination of niobium in tantalum-niobium alloys by means of oscillographic polarography. Ya. P. Gokhshtein, L. A. Genkina and A. M. Demkin (Inst. of Geochem. and Anal. Chem., Acad. Sci., USSR). *Zavod. Lab.*, 1959, **25** (9), 1042–1046.—The apparatus previously described (Gokhshtein *et al.*, *Zavod. Lab.*, 1959, **25**, 1008) is applied to the determination of Nb in 23 N, 15 N and 10 N H_2SO_4. G. S. SMITH

2176. Separation of niobium from tantalum in hydrochloric acid - oxalic acid medium by anion exchange. A. Speecke and J. Hoste (Lab. for Anal. Chem., Univ., Ghent, Belgium). *Talanta*, 1959, **2** (4), 332–340 (in French).—Niobium and Ta can be separated effectively by slow elution at 45° from an anion-exchange column (40 cm by 0·19 sq. cm, Dowex 1-X8, 100 to 200 mesh) with

either 0·01 M oxalic acid plus 2 M HCl (for removal of Ta) or 0·5 M oxalic acid plus M HCl (for removal of Nb). The method is useful for the removal of trace amounts of Ta from Nb or vice versa. The resin should contain ≃ 8% of divinylbenzene to ensure a high separation factor. Rapid elution at 25° is less satisfactory. W. J. BAKER

2177. Determination of tantalum in niobium with the Applied Research Laboratory's X-ray fluorescence monochromator. R. Burton, R. M. Jacobs and E. R. Valecko (Westinghouse Electric Corp., Bettis Plant, Pittsburgh, Pa.). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-617-1, July, 1958. 7 pp.—The method covers the concn. range 0·05 to 0·70%. The oxide - cellulose pellet is placed in the sample holder and the surface is irradiated. The fluorescent X-radiation from the pellet is dispersed and the integrated intensity of the analytical line is compared with that of a non-dispersed beam from an external standard. The recorded intensity ratios are referred to an appropriate analytical curve to obtain the concn. of Ta. NUCL. SCI. ABSTR.

2178. Determination of tantalum [in niobium pentoxide] by reaction with pyrogallol in the presence of tartaric acid. B. M. Dobkina and E. I. Petrova (State Sci. Res. Inst. of Rare Metals). *Zavod. Lab.*, 1959, **25** (9), 1064–1066.—The sample (0·3 to 0·5 g) is fused with 6 g of $K_4S_2O_7$, the melt is extracted with 100 ml of 10% tartaric acid soln. and the soln. is diluted to 250 ml with 0·1% tartaric acid soln. A 2-ml aliquot is mixed with 0·5 ml of 5% ammonium oxalate soln. and diluted with water to 3 ml. After 30 min., 5 ml of 8 M HCl and 2 ml of pyrogallol soln. (prepared by dissolving 20 g of pyrogallol in 75 ml of water, adding 10 ml of conc. HCl and 1 ml of freshly prepared 2 M SnCl₂, diluting to 100 ml and filtering) are added. The extinction is measured after 10 min. at 325 mμ. Corrections for the presence of Nb and Ti are made by means of aliquots of the same soln., the Nb being determined by means of thiocyanate and Ti by means of H_2O_2. G. S. SMITH

2179. Spectrographic determination of tantalum, titanium and zirconium in niobium. J. A. Featheringham, C. F. Lentz and R. M. Jacobs (Westinghouse Electric Corp., Bettis Plant, Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA-(GLA)-631-2, Aug., 1958. 4 pp.—The method is applicable to the analysis of metal samples that can be converted into the oxide by ignition. NUCL. SCI. ABSTR.

2180. Determination of tantalum as an impurity in zirconium, hafnium and niobium. R. S. Lauér and N. S. Poluéktov (Inst. of Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1959, **25** (8), 903–905.—With zirconium and hafnium samples the material (200 mg), in the presence of 20 ml of water, is treated dropwise with HF until it is dissolved. The soln. is evaporated to dryness, a further amount of HF is added, and the evaporation is repeated to ensure the removal of B. The residue is dissolved in 5 ml of water and the soln. is neutralised to methyl violet indicator with aq. NH₃ and shaken with 2 ml of 1·5 N HF, 5 ml of benzene and 1·8 ml of 0·2% methyl violet soln. The benzene extract is centrifuged and the extinction of the clear soln. is measured at ≃570 mμ. The method is suitable for samples containing ≃0·001% of Ta. With samples of niobium the material (10 mg) is

treated with 5 ml of HF followed by dropwise addition of HNO_3. After evaporation to dryness, two further evaporations with HF are carried out, and the residue is dissolved in 1·5 ml of HF and 5 to 6 ml of water. An aliquot (5 ml) of the diluted soln. (50 ml) is mixed with 4 ml of water, 10 ml of benzene and 1 ml of 0·2% methyl violet soln. The benzene extract is washed with 5 ml of 0·2 to 0·3 N HF containing 0·2% of methyl violet, and is then centrifuged and the extinction is measured. The methods are sensitive to 2·5 × 10⁻⁴ % of Ta in zirconium and hafnium and to 5 × 10⁻² % of Ta in niobium.   G. S. SMITH

2181. Spectrographic determination of the impurities titanium and tantalum in niobium pentoxide and titanium and niobium in tantalum pentoxide. N. I. Tarasevich and G. V. Kozireva. *Vestn. Moskov. Univ.*, 1959, (3), 185–188.—A spectrographic method is given for the determination of Ta (0·008 to 0·3%) and Ti (0·006 to 0·1%) in niobium pentoxide, with the line pairs Ti 2956·1 A - Nb 2955·46 A and Ta 2714·67 A - Nb 2711·86 A. Similarly the line pairs Nb 2950·88 A - Ta 2946·8 A and Ti 2956·1 A - Ta 2917·3 A are used for the determination of Nb (0·005 to 0·5%) and Ti (0·005 to 0·1%) in tantalum pentoxide. Standards are prepared by the use of pure metal oxides. The errors involved are 8 to 20% and depend on the concn.   A. BURWOOD-SMITH

2182. Isotopic analysis of oxygen in inorganic compounds. M. Anbar and S. Guttmann (Isotope Dept., Weizmann Inst. of Science, Rehovoth, Israel). *Int. J. Appl. Radiation and Isotopes*, 1959, **5** (3), 233–235.—Samples containing 0·2 to 0·3 millimole of O are heated in an ampoule at 400° for 2 hr. with 100 to 200 mg of $HgCl_2$ plus $Hg(CN)_2$ (1:1) at a pressure of 10⁻³ torr. The CO_2 produced is then condensed, by using liquid N, in a second ampoule containing 1·5 to 2 g of satd. zinc amalgam; after heating the ampoule for 2 hr. at 200°, the CO_2 is introduced into the mass spectrometer; if H is present at this stage it is pumped off after condensing the CO_2. The method has been applied to many oxides, barium phosphate, sulphate and arsenate, uranyl ferrocyanide and water (up to 200 mg). The results in 30 determinations on substances other than water were within ±10% of those by another method. The results in 13 determinations on water were within ±3% of those by the HOBr - Co method (*cf.* Anbar, *Anal. Abstr.*, 1959, **6**, 2859).   G. J. HUNTER

2183. New observations on the low-temperature reaction between hydrogen sulphide and sulphur dioxide. A novel method for analysing milligram quantities of hydrogen sulphide in the presence of carbon dioxide. I. K. Gregor and R. L. Martin (Sch. of Chem., Univ. of N. S. Wales, Broadway, Sydney). *Aust. J. Chem.*, 1959, **12** (3), 424–429.—A measured excess of SO_2 is condensed at −183° in a small evacuated tube (vol. ≃ 5 ml) together with the H_2S - CO_2 mixture to be analysed. The tube is sealed under high vacuum and then alternately warmed to room temp. and cooled to −183° several times to complete the reaction between the H_2S and the SO_2. The residual gas, containing CO_2 and unchanged SO_2, is fractionated under high vacuum through a Le Roy column (*Canad. J. Res., B*, 1950, **28**, 492) and the CO_2 and unchanged SO_2 are determined; H_2S may be determined from the fact that the reaction ratio between H_2S and SO_2 is 2·98:2.   C. A. SLATER

2184. Oxidation of sulphides with alkaline potassium permanganate. I. M. Issa and M. Hamdy (Fac. Sci., Cairo Univ., Giza, Egypt). *Z. anal. Chem.*, 1959, **169** (5), 334–339 (in English).—Sulphides are oxidised in alkaline $KMnO_4$ soln. to dithionates and this reaction is suitable for the determination of sulphides. The test soln. is mixed with an excess of 0·1 N $KMnO_4$, 0·2 g of telluric acid and sufficient 5 N NaOH to give a final soln. that is 0·2 N to 1·5 N with respect to the alkali. After the soln. has been allowed to stand for 5 min. it is poured into an ice-cold soln. of standard ferrous ammonium sulphate in 5 N H_2SO_4. The excess of Fe^{2+} is titrated with the standard $KMnO_4$ soln.   H. M.

2185. The problem of the membrane electrode. II. The use of "membrane voltammetry" for the precipitation titration of sulphate. P. Bersier, J. Bersier and F. Hügli (Abt. f. Chem., Technikum Winterthur). *Helv. Chim. Acta*, 1959, **42** (7), 2514–2518 (in German).—A method and apparatus are described and illustrated for the titration of SO_4^{2-} with $BaCl_2$ soln. with the use of an aluminium oxide membrane. At suitable c.d. (6·0 × 10⁻⁵ amp. per sq. cm of membrane) the potential (E) falls sharply at the end-point, from ≃ 3000 mV to ≃ 200 mV. The exact end-point is best found by plotting values of E against vol. in ml (v), readings being taken at 2-min. intervals, then plotting $\Delta E/\Delta v$ against v. The max. of the second curve is at the end-point. The method is also applicable to the titration of CrO_4^{2-} with Pb acetate and to SO_4^{2-} with Pb acetate.   H. M.

2186. Dissolution of barium sulphate by EDTA and sodium hydroxide. A. G. C. Morris (P.O. Box 21, Cato Ridge, Pietermaritzburg, Natal, S. Africa). *Chemist Analyst*, 1959, **48** (3), 76, 84.—The procedure of Belcher *et al.* (*Anal. Abstr.*, 1954, **1**, 448) whereby $BaSO_4$ is dissolved by boiling with a soln. of EDTA made alkaline with aq. NH_3 is modified by substituting an excess of 0·5 M NaOH soln. for the aq. NH_3. This, together with the addition of four times the theoretical amount of EDTA, is stated to reduce the time of dissolution from 6 hr. to 5 min. The solvent, however, extracts significant amounts of titratable metals from the glass apparatus used for the dissolution, and a Pyrex-glass or Vycor beaker should be used if an EDTA titration is to be carried out on the soln.   G. S. ROBERTS
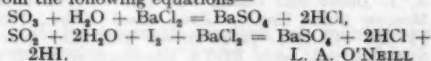
2187. Turbidimetric micro-determination of sulphate in plutonium solutions. H. D. Warren and A. Brunstad (General Electric Co. Hanford Atomic Products Operation, Richland, Wash.). *U.S. Atomic Energy Comm.*, Rep. HW-55349, March, 1958. 6 pp.—The basis of this procedure is the removal of Pu with a cation-exchange resin, followed by formation of a barium sulphate suspension and measurement of the absorption of the suspension with a spectrophotometer. The method can be used for the determination of sulphate in the concn. range 0·02 to 10 M in a 50-µl sample. With a larger sample, the lower limit could be extended. The coeff. of variation of a single result is ≃ ±3%; the accuracy, based on the analysis of known standards, is ≃ ±2·5%. Plutonium present in a 5-fold molar excess does not interfere.   NUCL. SCI. ABSTR.

2188. Extension of the Reich method. Determination of sulphur dioxide and sulphur trioxide in mixtures. E. Plana Eichberg (Tech. Sch., Bilbao,

Spain). *Inf. Quim. Anal.*, 1959, **13** (5), 135–137.—The Reich apparatus for the determination of SO_2 in gases, in which the gases are passed through a soln. of iodine until it is decolorised, has been modified with a device for sampling the gases under reduced pressure.   To analyse a mixture containing SO_2 and SO_3, the gases are passed through a soln. of iodine in aq. $BaCl_2$, and the total acidity is determined.   The concn. of SO_2 is determined in the usual way, and the SO_3 is calculated by difference from the following equations—

$$SO_3 + H_2O + BaCl_2 = BaSO_4 + 2HCl,$$
$$SO_2 + 2H_2O + I_2 + BaCl_2 = BaSO_4 + 2HCl + 2HI. \qquad \text{L. A. O'Neill.}$$

2189. Analytical applications of oxidations with ferricyanide catalysed by osmium tetroxide. III. Simultaneous determination of sulphur compounds. F. Solymosi and A. Varga. *Acta Chim. Acad. Sci. Hung.*, 1959, **20** (4), 399–405 (in English).—This is an English version of a paper published originally in Hungarian (*cf. Magyar Kém. Foly.*, 1959, **65**, 52; *Anal. Abstr.*, 1959, **6**, 4746).

P. D. Parr-Richard

2190. Titrations with quinol and similar reducing agents. XII. Determination of persulphates. O. Rottová and J. Zýka (Dept. of Anal. Chem., Charles' Univ., Prague). *Coll. Czech. Chem. Commun.*, 1959, **24** (8), 2809–2811 (in German).—Persulphates can be quant. reduced with an excess of standard quinol soln., with $AgNO_3$ as catalyst, the excess being determined with $Ce(SO_4)_2$ soln. *Procedure*—Dissolve the sample (10 to 50 mg) in H_2O (10 ml), add $AgNO_3$ soln. (0·25%) (0·5 to 1 ml), H_2SO_4 (20%) (5 ml) and 0·1 N quinol (5·0 to 10·0 ml) and heat; cool, add a few drops of ferroin as indicator and titrate with 0·1 N $Ce(SO_4)_2$.   The results agreed well with these by the iodimetric method; SO_4^{2-}, Cl^-, Br^-, NO_3^- and ClO_3^- did not interfere.

J. Zýka

2191. Determination of selenium and tellurium in gold-containing and platinum-containing slurries. A. N. Polukarov (Pyshminsk Electrolytic Copper Works). *Zavod. Lab.*, 1959, **25** (8), 905–909.—The sample (1 g) of a gold slurry containing, for example, Se 5·8%, Te 1·8%, Ag 30%, Au 2%, Sb 11%, As 2·9%, Cu 16% and Pb 12%, is dissolved in 50 to 60 ml of dil. HCl (2:1) with the addition of 2 ml of HNO_3, and the soln. is boiled for 1 to 2 min. with 0·5 to 1 g of hydrazine sulphate to precipitate Se. The cooled soln. is diluted with 100 to 120 ml of water to precipitate Au.   After 20 to 25 min. the ppt. is filtered off and washed with hot dil. HCl (1:30).   The filtrate is treated with 1 to 2 g of hydrazine sulphate and Te is pptd. by boiling the soln. (2·5 to 3 N in HCl) for 5 to 10 min.   The ppt. is washed with hot dil. HCl (1:30) and then dissolved in 25 to 30 ml of boiling dil. HCl (2:1) and 10 to 15 drops of HNO_3.   The soln. is diluted with hot water (150 to 200 ml) and treated with 2 g of urea, then cooled, and treated with 0·5 g of KI.   The iodine liberated is titrated with $Na_2S_2O_3$ soln. and the result is calculated to Te (%).   The ppt. containing Se, Au, AgCl, etc., is treated with dil. HCl and HNO_3 and the soln. is diluted with hot water to between 200 and 250 ml and treated with 2 g of urea.   To the cooled soln. a 1% soln. of mercaptobenzothiazole in 2 to 3% aq. NH_3 is added in $>30\%$ excess of the theoretical amount (2·54 g per 1 g of Au), and the ppt. of the gold complex and AgCl is filtered off after 5 to 15 min. and washed with hot dil. HCl (1:30).   The Se in the filtrate is deter-

mined by adding excess of $Na_2S_2O_3$ soln. and titrating the excess with iodine soln.   With platinum slurries containing, for example, Se 8%, Te 0·8%, Cu 25%, Ni 24%, Ag 4·7%, Au 0·5%, Sb 0·3%, Pb trace, Pd 5% and platinum metals 7·5%, the sample (1 g) is decomposed with 60 to 80 ml of dil. HCl (2:1) followed by 2 to 3 ml of HNO_3, and Se is pptd. by boiling the soln. with 1 to 1·5 g of hydroxyammonium chloride for 1 to 2 min.   The ppt. is collected and washed, and treated as described for gold slurries, except that 5 to 7 ml of the mercaptobenzothiazole soln. is sufficient for the pptn. of Pd and Au.   The soln. is boiled for 10 to 15 min. with 10 ml of HNO_3 to destroy hydroxyammonium chloride and the soln., at 40° to 50°, is treated with 5 to 10 ml of 30% $FeCl_3$ soln. and aq. NH_3.   The ppt. is collected and washed with dil. aq. NH_3, and re-dissolved in hot dil. HCl (1:2).   By addition of $SnCl_2$ to the soln. at 80° to 90°, Te is pptd. and is then determined as described above for gold slurries.

G. S. Smith

2192. An EDTA - polarographic method for the determination of tellurium in lead alloys. Z. P. Zagorski and M. Cyrankowska (Central Lab. of Accumulators and Cells, Poznan, Poland). *Talanta*, 1959, **2** (4), 367–369 (in English).—From 0·01 to 0·1% of Te in a lead alloy can be determined rapidly by recording the polarogram obtained with a soln. of the sample in HNO_3 (1:1), containing EDTA, NH_4Cl and NH_4NO_3, and adjusted to a pH of 8 to 9 with aq. NH_3; a mercury-pool anode is used.   The soln. should be de-aerated with a stream of propane.   The Te^{IV} - Te wave has $E_{\frac{1}{2}} = -0.45$ V.   The error is 2·4% at the level of 0·1% of Te.   There is interference by As, Sb and Tl when the concn. of each is $>0.01\%$, but a correction can be made for Tl.

W. J. Baker

2193. Determination of tellurium in the presence of antimony in alloy amounts, using EDTA. Z. P. Zagorski and M. Cyrankowska (Central Lab. of Accumulators and Cells, Poznan, Poland). *Talanta*, 1959, **2** (4), 380 (in English).—The turbidimetric method of Johnson and Anderson (*Anal. Chem.*, 1955, **27**, 120) can be extended to the determination of Te in tellurium - antimony - lead alloys. *Procedure*—Dissolve the sample (1 g) in 50 ml of conc. HCl containing Br, boil to expel Br, and then make the soln. up to 100 ml with HCl (2:1). Evaporate an aliquot (25 ml) to $\simeq 3$ ml, add 10% EDTA soln. (5 ml), 4% gum acacia soln. (6 ml) and H_2O (40 ml).   Heat to dissolve any ppt. and add 5 ml of a soln. of NaH_2PO_2 [32 g in 100 ml of HCl (1:1)].   Heat for 15 min., cool and adjust to pH 1 to 2 with aq. NH_3.   Make up to 100 ml and measure the extinction at 435 mμ.   The error is $\simeq 5\%$.

W. J. Baker

2194. Spectrographic analysis of chromium for impurities. E. V. Lifshits and N. I. Bugaeva (Phys.-Tech. Inst., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1959, **25** (8), 952–954.—For less volatile impurities, direct fractional evaporation in a d.c. arc is used; for the more volatile impurities, enrichment by the evaporation method at 1500°, followed by excitation in a condensed spark, is used.

G. S. Smith

2195. Separation of chromic acid with the aid of ion exchangers: limits of resistance. R. Weiner and C. Schiele (Lab. for Electron Microscopy, Univ. Innsbruck, Austria). *Z. anal. Chem.*, 1959, **169** (4), 271–276 (in German).—Solutions for analysis are

freed from CrO_3 by passage over Amberlite IRA-410. (Cl⁻ form) which is stable in 1% aq. CrO_3 soln. for >30 min. and is only slightly attacked by concn. >1·5%. Hold-up is very low (usually <1%).

　　　　　　　　　　　　　　　　J. P. STERN

2196. Potentiometric titration of molybdenum(V) with ferric alum. G. Gopala Rao, Seetharamaraju Sagi and M. Suryanarayana (Dept. of Chem., Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1959, **169** (4), 248–253 (in English).—The reaction of Mo^V with Fe^{III} is slow at room temp., but at 98° to 100° in 0·5 to 1·0 N HCl it is fast enough for use in potentiometric titration in an inert atmosphere. At higher acid concn. the potential jump at the end-point is reduced or does not occur. By this method Mo^V (0·3 to 0·6 millimole) can be determined with an accuracy within ±0·3%.　　J. P. STERN

2197. Oxidimetric methods for the volumetric determination of molybdenum(V). I. Titration with ceric sulphate and sodium vanadate. Study in the mechanisms of redox indicator reactions. G. Gopala Rao and M. Suryanarayana (Dept. of Chem., Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1959, **169** (3), 161–169 (in English).—The titration of Mo^V with $Ce(SO_4)_2$ soln., with ferroin as indicator, is best carried out in 3 N HCl; with diphenylbenzidine as indicator, a medium that is at least 4 N with respect to HCl is required. In both cases the addition of 5 ml of syrupy H_3PO_4 is recommended for every 50 ml of reaction mixture. In the titration of Mo^V with $NaVO_3$ soln., with N-phenylanthranilic acid as indicator, an acid concn. of at least 6 N is required at the end-point of the titration; a concn. of 4 N is satisfactory if oxalic acid is added. When diphenylbenzidine is used as indicator the medium must be 4 N in HCl and either oxalic or phosphoric acid must be present. If both acids are added the titration can be conducted in 3 N HCl.　　　　　　G. P. COOK

2198. Determination of molybdenum in alloys with 8-mercaptoquinoline. R. B. Golubtsova (A. A. Balkov Inst. of Metallurgy, Acad. Sci. USSR, Moscow). *Zhur. Anal. Khim.*, 1959, **14** (4), 493–496.—To determine Mo in chrome-nickel alloy, dissolve 0·01 g of alloy in 5 ml of conc. HCl and 5 drops of conc. HNO_3; add 1·5 ml of conc. H_2SO_4, evaporate (×3) to white fumes, and cool. Add 1·5 ml of water and, if the alloy contains W, 1 ml of conc. H_3PO_4, and warm. Make the soln. up to 100 ml and to a 1 to 3-ml aliquot add, in the following order, 2·5 N HCl (2 ml), water (2 ml), 10% ascorbic acid soln. (1 ml) (or, if W is present, 1 ml of conc. H_3PO_4) and 1 ml of 5% 8-mercaptoquinoline soln. (5 g of reagent dissolved in 25 ml of conc. HCl and 75 ml of water). Mix the soln. and extract the Mo by shaking with 2 ml of toluene for 3 min. The Mo is determined either by comparing the colour with a standard series or spectrophotometrically. For the latter method use 5 ml of the test soln. and 5 ml of toluene, and measure the extinction of the extract at 420 mμ. A calibration curve is constructed by adding known amounts of Mo to soln. obtained from the alloy. To determine Mo in titanium-base alloys, dissolve 0·01 g of alloy in 5 ml of conc. H_2SO_4, add 7 drops of conc. HNO_3 and evaporate (×3) to SO_3 fumes. Cool, add 1·5 ml of conc. H_3PO_4 (for ≯5% of W) or 1·25 ml of 5% succinic acid (for >5% of W), make up to 100 ml and proceed as described above. The detectable minimum is 0·2 μg of Mo at a limiting dilution of 1 in 1 × 10⁶. Soln. containing

1 to 10 μg of Mo per ml obey Beer's law. In determining 0·6 to 5·2% of Mo in a nickel-base alloy containing ≯10% of W, the maximum error was 6% (relative). In determining 0·65 to 8·2% of Mo in a titanium-base alloy containing 1·5% of Cr and ≯20% of W, the maximum error was 2·5% (relative).　　　　　　　　　C. D. KOPKIN

2199. Complexometric determination of molybdenum after its reduction to the quinquevalent state. A. I. Busev and Chang Fan (M. V. Lomonosov Moscow State Univ.). *Zhur. Anal. Khim.*, 1959, **14** (4), 445–450.—It is shown that Mo^V reacts quantitatively with EDTA (disodium salt) (I) in the molar ratio 2:1 to form a complex over a wide range from pH 10 to 0·5 M HCl. To determine Mo, add to a soln. of hydrazine hydrochloride (0·01 g per ml) (2 to 5 ml), in a 200-ml beaker, conc. HCl (1 to 3 ml) and water to 30 ml; heat to boiling and add dropwise not more than 10 ml of the neutral or slightly acid molybdate test soln. (containing 10 to 100 mg of Mo) and continue boiling for several minutes till the soln. becomes yellow-brown. Add a known vol. of 0·01 M I and Eriochrome black T indicator. Neutralise the soln. with aq. NH_3 or NaOH soln. till the colour changes from yellow to pale green and, for each 100 ml of soln., add 2 ml of a NH_4Cl - aq. NH_3 buffer soln. of pH 10. Boil the soln., cool to between 40° and 60° (when the soln. should be clear green in colour; if not add a little more indicator) and titrate with 0·01 M $ZnSO_4$ to a red - brown colour. In determining 9·5 to 114 mg of Mo, the max. error is 1%; with smaller amounts of Mo the error increases. Tartrates, citrates, oxalates, nitrates, nitrites, oxidising agents and Fe interfere. Tungsten (≯10 mg in 40 ml of soln.) does not interfere. Elements normally titrated with I in alkaline medium interfere; in this case it is possible to titrate total interfering elements together with Mo^V, then the interfering elements without reducing Mo^{VI}, which does not form a stable complex with I under the titration conditions; Mo is then found by difference. In the presence of Mg and Ca use $MgSO_4$ for the titration and use NaOH or KOH for the neutralisation.　　　　　　C. D. KOPKIN

2200. Cerimetric determination of molybdenum in high chloride media using the molybdenum blue reaction. C. M. Callahan, S. C. Foti and J. R. Lai (Naval Radiological Defense Lab., San Francisco). *U.S. Atomic Energy Comm.*, Rep. USNRDL-TR-362, Sept., 1959. 13 pp.—After passage through a Jones reductor the reduced molybdenum is collected under a soln. of sodium molybdate, the pH of which is so adjusted that an equivalent amount of molybdenum blue (I) is formed, which is then titrated to a colourless end-point with standard $Ce(SO_4)_2$ soln. The formation of I serves two purposes—the oxidation - reduction reaction between Mo^{III} and Mo^{IV} forms I as an intermediate oxidation state that is stable against air oxidation, and the I serves as its own indicator in the titration. The results obtainable by this method agree with those obtained by the use of permanganate in H_2SO_4.

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2201. Polarographic determination of molybdenum in acid solutions. Pei-Tse Lee, Teh-Heng Chao and Yu-Shien Lee. *Acta Sci. Nat.*, 1959, (1), 99–104.—At H_2SO_4 concn. of 4 M and acetic acid concn. of 0·1 M, three waves due to Mo are distinctly visible and there are no maxima. The

$E_{\frac{1}{2}}$ for the third wave is -0.23 V. The concn. range favourable for the determination is 10^{-5} to 10^{-3} M. The effects of various foreign ions (e.g., Mn^{2+}, Zn^{2+} and Cr^{3+}) have been studied. In general, these elements do not interfere. The results of the polarographic determination of Mo in molybdenum ores are in good agreement with those obtained either by thiourea - molybdenum thiocyanate colorimetry or by using a cupferron reagent.

SCI. ABSTR. CHINA

2202. Spectrographic analysis of molybdenum metal powder. R. Dyck and T. J. Veleker (Chem. and Metall. Div., Sylvania Electric Products Inc., Towanda, Pa., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1640–1643.—Sixteen elements, commonly occurring as impurities in molybdenum, are determined. These are divided into four groups—(*i*) Cr, Fe, Mn, Ni, Pb, Si and Sn; (*ii*) Al, Ca, Cu and Mg; (*iii*) Ba, K, Na and Sr; and (*iv*) W, and conditions for the determination of each group are given. For groups (*i*) and (*iii*), molybdenum powder (1 g) is mixed with a buffer [25 parts of SP2 graphite and 1 part of cobalt (< 200 mesh)] (0.28 g). For group (*ii*), a similar procedure is used, but with nickel in place of cobalt. For tungsten, the sample is mixed with an equal bulk of ZnO. A graphite cup-electrode is used. For group (*i*), the excitation conditions are a high-voltage a.c. arc, a Littrow spectrograph in the range 2400 to 3200 A, and the Co 2685·3-A line as internal standard; for group (*ii*) a high-voltage a.c. arc, a Littrow spectrograph in the range 3200 to 4100 A and Ni 3080·3 A as internal standard; for group (*iii*) a dual-grating instrument (15,000 and 30,000 lines), a d.c. arc, and superimposed spectra (5725 to 7625 A and 4000 to 5000 A), with Co 7085·0 A as internal standard; for group (*iv*), the dual-grating instrument (15,000 set for 7000 to 9000 A) with a d.c. arc. The W lines (4008·7 and 4102·7 A) are evaluated against Mo 4006·0 A. The ranges covered are of the order 0·0005 to 0·05%, except for W (0·01 to 1·0%) and Mn, Cu, Ca and Mg (0·0001 to 0·01%). Reported coeff. of variation range from ± 7 to ± 17%.   T. R. ANDREW

2203. The potentiometric reduction of solutions containing molybdenum and tungsten ions. H. K. El-Shamy and M. F. Barakat (Chem. Dept., Fac. Sci., Alexandria Univ., Egypt). *Egypt. J. Chem.*, 1959, **2** (2), 191–205 (in English).—Optimum conditions have been established for the volumetric determination of Mo^{VI} or W^{VI} by titration with $CrCl_2$, according to the procedure described for U^{VI} by El-Shamy and El-Din Zayan (*Analyst*, 1955, **80**, 65); the soln. should be 8 M in HCl and 0·2 M in oxalic acid and the titration is carried out at 85°. Results for Mo may be obtained at HCl concn. $\not< 2$ M, but below this level the titration curves always show a steep initial drop in potential, and only one point of inflection corresponding to the $Mo^{VI} \rightarrow Mo^{V}$ end-point; the higher acidities give a well-defined inflection for $Mo^{V} \rightarrow Mo^{III}$. Mixtures of Mo and W give only two inflections, the second corresponding to $Mo^{V} \rightarrow Mo^{III}$ and $W^{VI} \rightarrow W^{V}$. Results of analyses of soln. containing binary mixtures of Mo^{VI}, W^{VI} and U^{VI} at the 0·5-millimole level are said to be within ± 0.5% of the amount added. The examination of a ternary mixture was unsatisfactory. Molybdophosphoric acid and molybdosilicic acid could be successfully titrated directly in 8 M HCl - 0·2 M oxalic acid at 85°, but the corresponding tungsten compounds were not reduced.   T. R. ANDREW

2204. Spectrographic determination of tungsten in niobium. J. A. Featheringham, C. F. Lentz and R. M. Jacobs (Westinghouse Electric Corp., Bettis Plant, Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-631–5, Aug., 1958. 3 pp.—The method is applicable to the analysis of metal samples that can be converted into the oxide by ignition.   NUCL. SCI. ABSTR.

2205. Chemico-spectrographic method for determining tungsten in molybdenum. N. I. Tarasevich, A. D. Khlýstova and E. A. Pak (Moscow State Univ.). *Zavod. Lab.*, 1959, **25** (8), 955–956.—Enrichment is carried out on the soln. of the sample by co-pptn. of W with ammonium molybdophosphate. Standards are treated similarly. The content of W in the ppt. is determined by means of an a.c. arc.

G. S. SMITH

2206. Determination of uranium by the fluoride method with a titrimetric finish. V. M. Evenigorodskaya and M. I. Ryanicheva. *Zhur. Anal. Khim.*, 1959, **14** (4), 457–462.—Decompose the sample (0·2 to 1 g) with a mixture of conc. HNO_3 and conc. HCl, remove excess of acids by evaporation, add 23 to 25 ml of H_2SO_4 (1:1) and heat to dense white fumes. Add 50 or 60 ml of water and boil for 20 to 30 min. to dissolve the salts. Transfer the soln. and residue to a flask, make up to 200 ml, remove a 25- or 50-ml aliquot and add the following reagents, to give concn. in the final volume of 100 ml, of N H_2SO_4, 2% of $(NH_4)_2SO_4.FeSO_4.6H_2O$, 2% of Na_2SO_4 and 10% (by vol.) of 40 to 48% HF. Mix the soln. well and set aside for 5 hr. or overnight. Filter through a waxed funnel, wash the ppt. of $NaUF_5$ with a wash soln. of pH $\simeq 2$ (50 ml of HF and 40 g of ammonium acetate made up to 1 litre) until no reaction for Fe^{3+} is given. Transfer the filter and ppt. to a 250-ml flask containing 90 ml of 33% (v/v) acid, 10 ml of satd. H_3BO_3 soln. and 2 or 3 drops of 0·1% phenylanthranilic acid soln., mix well and titrate with 0·01 or 0·005 N ammonium vanadate to a stable pink - violet colour. Niobium, Ta, Mo and Ti remain in soln. when the $NaUF_5$ is pptd.; Mo forms molybdenum blue, which can be almost completely washed out of the ppt.; Fe^{III}, Al, Ca and the rare-earth elements are partially pptd., but do not interfere in the titration. Uranium (3 to 60%) has been determined with a max. error (relative) of 2·8%.   C. D. KOPKIN

2207. Spectrophotometric study of uranium solutions. II. Spectrophotometric determination of uranium in ores and minerals as thiocyanate after extraction with ethyl methyl ketone. S. I. Sinyakova and N. S. Klassova (V. I. Vernadskii Inst. Geochem. and Anal. Chem., Acad. Sci. USSR, Moscow). *Zhur. Anal. Khim.*, 1959, **14** (4), 451–456.—After extraction of uranyl nitrate with ethyl methyl ketone (**I**) from a soln. satd. with NH_4NO_3, the extinction curve of the thiocyanate complex of U in a medium of acetone - **I** (3:2) is very similar to that in water - acetone (2:3), the extinction coeff. being slightly greater. Ferric iron, Cu^{2+}, Co^{2+}, Bi and Ti^{IV} are partially extracted with **I** and cause high results; V also causes high results, and Mo low results. To determine U in the absence of Mo, V, Ti and Bi, but in the presence of Fe, Cu and Co, extract U as uranyl nitrate with **I** (2 $\times$ 2 ml) from a soln. satd. with NH_4NO_3, collect the extracts in a 10-ml flask, add 6 ml of a satd. NH_4SCN soln. in acetone, make up to volume with **I**, and measure the extinction at 350, 375 or 400 mμ after adding a few crystals of

ascorbic acid to mask Fe^{III}, Cu^{2+} and Co^{2+}. The relative error is 2%. In the presence of Mo, Fe, Cu and Co, but in the absence of V and Ti, proceed as described above, but add 1 ml of 40% lactic acid soln. to the soln. before the extraction, to complex Mo. The accuracy is the same. In the presence of V, Fe, Co and Cu, but in the absence of Mo, proceed as described above, but add 1 ml of 4·7% $Zr(NO_3)_4.5H_2O$ soln. before the extraction. In the presence of Fe, Cu, Co, V and Mo, re-extract the U from the I layer with 5 ml of 2% ammonium carbonate soln. ($\times 3$), evaporate the soln. to dryness, ignite the residue for 10 to 15 min. between 500° and 600°, cool, dissolve it in conc. HNO_3 (2 to 3 ml), evaporate to dryness, dissolve the salt in 9 ml of satd. NH_4NO_3 soln., add 1 ml of the 4·7% zirconium nitrate soln., extract twice with I and continue as described above. Construct calibration curves. Beer's law is obeyed in the concn. range studied (4.2×10^{-5} to $6.3 \times 10^{-4}\,M$ in U). Uranium (0·01 to 1·0%) in ores and minerals can be determined with an error (relative) of $\pm 3\%$.

C. D. KOPKIN

2208. Determination of uranium by square-wave polarography. G. W. C. Milner and J. H. Nunn (Anal. Chem. Group., A.E.R.E., Harwell, Berks., England). *Anal. Chim. Acta*, 1959, **21** (3), 266–275 (in English).—An examination of a series of mineral-acid supporting-electrolytes, with and without added tartrate, indicated that a soln. containing perchloric acid and tartrate gave a linear calibration graph over the range 0·6 to 50 μg of U per ml. Molybdenum, V and Cr interfere at high ratios to U, as do Bi, Cu and Fe, although less seriously; Zn, Ni, Cd, In, Co and Mn are without effect. *Procedure*—Heat the sample (100 mg) with Na_2O_2 (1·0 g) at 480° for 20 min. Dissolve the product in water, add conc. HCl (10 ml) and evaporate to dryness. Bake for 20 min. on a hot-plate, add conc. HCl (20 ml) and water (20 ml). Filter, wash the residue with dil. HCl and dilute the filtrate and washings to 100 ml. To a 5-ml aliquot add 10 N $HClO_4$ (1 ml), heat to fumes, cool, add tartrate soln. (120 g of Na tartrate and 3 g of NaCl per litre) (0·5 ml), and dilute to 5 ml. De-aerate and record the uranium peak on a square-wave polarograph, scanning from -0.1 to -0.6 V *vs.* the mercury-pool anode. Repeat the scan after making a standard addition of U. The method is illustrated by the analysis of several minerals containing known quantities of U in the range 0·02 to 0·3%.

W. T. CARTER

2209. Polarographic determination of small amounts of uranium in phosphates with ion exchangers. F. Habashi (Inst. für anorg.-chem. Technol., Tech. Hochsch., Wien). *Mikrochim. Acta*, 1959, (6), 932–941 (in German).—The procedure of Fisher and Kunin (*Anal. Abstr.*, 1957, **4**, 2970), in which the addition of H_2SO_4 to the H_3PO_4 soln. converts the U into an anionically-charged uranyl sulphate complex, which is held on Amberlite IRA-400, is adapted for the preliminary separation of U from H_3PO_4 and other impurities. Microcolumns with a capacity of approx. 0·75 cu. cm are used. Uranium is eluted with N HCl, the eluate is evaporated to dryness, and H_2SO_4 is fumed off. The residue is dissolved in 0·01 N HNO_3 and polarographed.

D. F. PHILLIPS

2210. Influence of phosphoric acid on the determination of uranium with ammonium thiocyanate. F. Habashi (Inst. für anorg.-chem.

Technol., Tech. Hochsch., Wien, Austria). *Talanta*, 1959, **2** (4), 380–382 (in German).—In the colorimetric determination of U (0·1 to 0·4 mg per ml) with thiocyanate, the intensity of the coloration decreases with increasing concn. of H_3PO_4 (1 to 8 M). At concn. of H_3PO_4 of 4 to 8 M the colour is unstable, rapidly becoming reddish, especially when the extinction of the blank is being measured against water. When present at these higher concn. PO_4^{3-} should previously be removed, and at lower concn. all optical measurements should be made in a colorimeter fitted with a blue filter.

W. J. BAKER

2211. Determination of uranium and molybdenum by selective precipitation with 8-hydroxyquinoline. L. E. Storms (Goodyear Atomic Corp., Portsmouth, Ohio). *U.S. Atomic Energy Comm.*, Rep. GAT-285, July, 1959. 13 pp.—The precipitation is carried out in the presence of EDTA (disodium salt). The U is pptd. from an ammoniacal soln. containing Mo, and the Mo is pptd. from the filtrate by adjusting the pH with acetic acid after the addition of ammonium acetate. The procedure was found to be applicable to the determination of U and Mo in the soln. resulting from the hydrolysis of the hexafluorides. For an approx. equimolar mixture of the hydrolysed hexafluorides, a precision of ± 0.14 mole % of molybdenum hexafluoride (I) can be expected. The limit of error for the mean of three determinations will be ± 0.08 mole % of I. The absolute error in determining the mole % of I is estimated to be $\pm 0.25\%$.

NUCL. SCI. ABSTR.

2212. The optical spectra of some rare-earth and transuranic elements in the 1 to 3-micron region. IV. The spectrum of neptunium. L. Bovey and E. B. M. Steers (Chem. Div., A.E.R.E., Harwell, Berks., England). A.E.R.E. Report AERE-R 3118, 1959. 5 pp.—A list of wavelengths and intensities for neptunium lines is given, together with a short discussion of the experimental technique.

2213. Determination of small amounts of neptunium in plutonium metal. L. J. Slee, G. Phillips and E. N. Jenkins (Anal. Chem. Group, A.E.R.E., Harwell, Berks., England). *Analyst*, 1959, **84**, 596–603.—The sample is dissolved in HCl containing hydroxyammonium chloride (to reduce Pu to Pu^{III}), and the Np is reduced to Np^{IV} with $FeCl_2$ and extracted quant. with a soln. of 2-thenoyltrifluoroacetone in xylene. The Np is extracted from the organic phase with HNO_3, residual organic matter is removed by a wet-ashing procedure, the residue is dissolved in HCl, the vol. of the soln. is reduced by evaporation with hydroxyammonium chloride, EDTA (disodium salt) soln. is added, the pH is adjusted to between 5·5 and 6·5, and the Np is determined by square-wave polarography. The method is applicable to the determination of 10 to 2000 p.p.m. of Np. The precision is $\simeq \pm 10\%$ and $\pm 2\%$ at the levels of 25 and 500 p.p.m., respectively.

A. O. JONES

2214. Analytical procedures for the plutonium metal fabrication process. VI. The copper spark method. T. K. Bierlein, L. F. Kendall and H. H. Van Tuyl (Hanford Works, Richland, Wash.). *U.S. Atomic Energy Comm.*, Rep. HW-25035, July, 1952. Decl. July, 1959. 24 pp.—In the copper spark method for the determination of trace impurities, the sample, in HCl soln., is evaporated on copper electrodes which are sparked to produce the spectra of the impurities. Investigations of

the variables affecting sensitivity, electrode preparation, excitation conditions and the evaluation of the spectra are discussed. With very careful control of all the variables, precisions of $\simeq \pm 2\%$ at the 99% limits can be obtained. A stepwise procedure for the semi-quant. analysis of soln. containing small amounts of Pu is included.

VII. Cupferron extraction - copper spark method. T. K. Bierlein, L. F. Kendall and H. H. Van Tuyl. *U.S. Atomic Energy Comm.*, Rep. HW-25074, July, 1952. Decl. July, 1959. 23 pp.—Ter- or quadri-valent plutonium is separated from impurity elements by extraction of the plutonium cupferride into $CHCl_3$. The aq. phase, containing cobalt as internal standard, is evaporated to dryness and placed on copper electrodes which are sparked to produce the impurity spectrum. The amounts of the impurities present are determined by comparison of the densities of impurity lines with standard spectra and correcting for the recovery of the cobalt internal standard. NUCL. SCI. ABSTR.

2215. Spectrochemical determination of trace impurities in plutonium nitrate solutions. A. J. Johnson and E. Vejvoda (Rocky Flats Plant, The Dow Chemical Co., Denver, Colo., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1643-1646.—A soln. of NaF (4%) (25 μl) is evaporated to dryness in the shallow crater of a waterproofed graphite electrode (UCP 4925) and 25 μl of the sample soln. (containing 500 μg of Pu) is subsequently evaporated on the residue. The treated electrode is exposed in a 3·4-metre grating spectrograph (15,000 lines per in.) over the range 2100 to 4100 A, under the following conditions—2400 V, 4·5 amp., a.c. arc, 4 sec. pre-burn, 4 sec. exposure with a 4-mm analytical gap, 30-micron slit width, and 1·5-mm slit height. Analytical wavelengths are reported for 34 elements. The lower limits of detection range from 4 to 10,000 p.p.m.   T. R. ANDREW

2216. Volumetric determination of fluorine in tetrafluoroborates. Z. T. Maksimýcheva and V. Khakimova (V. I. Lenin Central Asia State Univ.). *Zavod. Lab.*, 1959, **25** (8), 911-913.—The soln. (containing between 15 and 29 mg of F in 100 ml) is boiled under reflux with 5 ml of 2 N HNO_3 for 1 hr., a few drops of bromophenol blue indicator soln. and 3 ml of 10% NaCl soln. are added, and the soln. is neutralised by means of dil. HNO_3 and dil. NaOH soln. The blue soln. is treated with 2 ml of dil. HCl (1 : 1) and 20 ml of 25% $Pb(NO_3)_2$ soln., and then with 5 g of Na acetate, heated on a boiling-water bath for 30 min., and set aside overnight at room temp. The ppt. is collected and washed, once with cold water, then four times with saturated PbClF soln. and again with cold water, and then dissolved in 20 ml of hot 25% HNO_3. The soln. is treated with an excess of 0·1 N $AgNO_3$ and heated for 30 min. The cooled soln. is filtered, and the filtrate is titrated with NH_4SCN soln. in the presence of Fe^{3+}.   G. S. SMITH

2217. Voltammetric, potentiometric and amperometric studies with a rotated aluminium wire electrode. III. Amperometric determination of fluoride with the R. Al. E. I. M. Kolthoff and C. J. Sambucetti (School. of Chem., Univ. of Minnesota, Minneapolis, U.S.A.). *Anal. Chim. Acta*, 1959, **21** (3), 233-245 (in English).—Fluoride in water, in the range 0·2 to 6 p.p.m., may be determined by measuring the anodic current at the rotating

aluminium electrode at -0.75 V *vs.* the S.C.E. Both the sensitivity of the electrode and the residual current increase with decreasing pH, and the optimum value is considered to be 3·0 to 3·6. Low concn. of nitrate, chloride, sulphate and the alkaline-earth metals are without effect, but phosphate and EDTA cause a reduction in sensitivity; Al interferes seriously, but can be masked by EDTA; oxidising agents must be absent. The method of standard addition is used to overcome the varying reproducibility of the electrode. With 20 samples of known fluoride content over the range 0·2 to 2 p.p.m. the method gave results with a maximum error of 10%.   W. T. CARTER

2218. Indirect argentimetric and mercurimetric determination of the fluoride ion with potentiometric or redox end-point indication. J. Bognár and L. Nagy (II Chem. Dept., Tech. Univ. for Heavy Industries, Miskolć, Hungary). *Magyar Kém. Foly.*, 1959, **65** (9), 335-341.—An improved method based on the pptn. of PbClF (I) is described. After dissolving the pptd. I, Cl^- (or F^-) are determined potentiometrically; the calomel electrode can be replaced by a simple silver chloride - copper amalgam electrode (prep. described). Under suitable conditions of pptn. the errors of the macro, semi-micro and micro procedures are $<0.1\%$, $<0.2\%$ and $<1\%$, respectively. In a modification of the method, I is dissolved in 4 N NaOH, the soln. is acidified with conc. H_2SO_4, and Cl^- or F^- are titrated with $Hg(NO_3)_2$ soln. in the presence of $K_3Fe(CN)_6$, with Setoglaucin O (C.I. Basic Blue 1) or Astra blue G as indicator. This procedure is suitable for the semi-micro or micro scale only; the errors are $\pm 0.4\%$ and $\pm 1\%$, respectively.

A. G. PETO

2219. Analytical studies on fluoride. IV. Determination of a small amount of fluorine in rocks. Koichi Emi, Tadashi Hayashi and Hiroshi Takeda (Fac. of Sci., Okayama Univ., Tsushima, Okayama). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (7), 731-735.—For the separation of F from Si, the ZnO - Na_2CO_3 fusion method of Grimaldi *et al.* (*Anal. Chem.*, 1955, **27**, 918) was adopted, with sodium aluminate soln. in place of water for the extraction of F^- from the melt. The Al does not interfere with the distillation of HF from a mixture of H_2SO_4 and H_3PO_4. The HF is collected in 0·1 N NaOH and determined colorimetrically with arsenazo (Emi and Hayami, *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1955, **76**, 1291). *Procedure*—Fuse the sample (0·5 g) with ZnO (0·5 g) and Na_2CO_3 (10 g) for 1·5 hr. and dissolve the melt in sodium aluminate soln. [1 g of $Al_2(SO_4)_3$ in 100 ml of 3% NaOH soln.] (20 ml), filter and wash the ppt. with water. Dilute the filtrate to $\simeq 70$ ml, add Ag_2SO_4 (a few mg), powdered glass (1 g), H_2SO_4 (sp. gr. 1·84, 50 ml) and H_3PO_4 (sp. gr. 1·7, 10 ml) and steam-distil at 135° at a rate of $\simeq 5$ ml per min. Collect $\simeq 200$ ml of distillate in 0·1 N NaOH (25 ml), neutralise to *p*-nitrophenol and make up to 250 ml for photometry.

V. Determination of fluorine in animal organs. Koichi Emi, Tadashi Hayashi and Hiroshi Takeda. *Ibid.*, 1959, **80** (7), 735-738.—The organ is dried at 120°, calcined with CaO (15% of the dry weight of the whole organ) at 350° to 450° and ignited at 700° to 750° for 30 min. The ash is distilled with acid and the F is determined as previously described (Emi and Hayakawa, *Anal. Abstr.*, 1958, **5**, 2726).

K. SAITO

2220. Colorimetric determination of chloride in concentrated hydrogen peroxide. I. Geld and I. Sternman (Metal Chemistry Sect., U.S. Naval Material Lab., New York, N.Y.). *Anal. Chem.*, 1959, **31** (10), 1662–1664.—Decompose the sample (50 ml) by heating it with de-ionised water (10 ml) and NaOH soln. (6 N) (1·0 ml) in a 500-ml beaker at 75° to 90°, then evaporate the soln. to dryness on a steam bath. Dissolve the residue in 1 to 2 ml of de-ionised water and add HClO$_4$ (70%) (0·6 ml) and reagent soln. [1 vol. of aq. Hg(SCN)$_2$ soln. satd. at 25° and 2 vol. of a soln. made by dissolving 14 g of iron wire in dil. HNO$_3$, evaporating to fumes with 120 ml of HClO$_4$ (70%) and diluting to 1 litre] (3 ml). Transfer to a 10-ml flask, dilute to volume, and read the extinction against water at 460 mμ. The range covered is from 0·1 to 40 μg of Cl$^-$ per litre of 90% H$_2$O$_2$, with a precision of 0·1 mg per litre or better. Potentially interfering ions are normally present in concn. too small to affect results.   T. R. ANDREW

2221. New colorimetric determinations by the use of thiocyanate. XV. Determination of a small amount of bromide in the presence of chloride and iodide. Iwaji Iwasaki, Satori Utsumi, Akihisa Tomonari, Ichiro Morita and Masaru Shiota (Lab. of Anal. Chem., Inst. of Tech., Ookayama, Meguro-ku, Tokyo). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (7), 744–749.—Bromide (<50 p.p.m.) in natural water (10 ml) is oxidised to bromine with KMnO$_4$ (0·02 M, 0·7 ml) and H$_2$SO$_4$ (18 N, 0·3 ml) in <5 min. and is extracted with CCl$_4$ (2 × 5 ml). Under these conditions iodide (<20 p.p.m.) is oxidised to IO$_3^-$ and Cl$^-$ (<1%) remains unchanged. The CCl$_4$ soln. is washed with N H$_2$SO$_4$ (5 ml), shaken with an aq. soln. containing (NH$_4$)$_2$SO$_4$.Fe(SO$_4$)$_3$ and Hg(SCN)$_2$, and the extinction of the aq. layer is measured at 460 mμ after 10 min. (Utsumi *et al.*, *Ibid.*, 1952, **73**, 889). There is no interference from ions common in natural water. For <6 p.p.m. of Br$^-$, the amounts of sample, 18 N H$_2$SO$_4$, 0·02 M KMnO$_4$ and CCl$_4$ used are increased to 50, 1·5, 2·5 and 10 ml, respectively.

XVI. Determination of a small amount of iodide in the presence of chloride and bromide. Iwaji Iwasaki, Satori Utsumi, Akihisa Tomonari and Ichiro Morita. *Ibid.*, 1959, **80** (7), 749–753.—Iodide (<60 p.p.m.) in natural water is oxidised to iodine in the presence of H$_2$SO$_4$ (18 N, 0·5 ml) and H$_2$O$_2$ (3%, 1 ml) in <5 min. and is extracted with CCl$_4$ (2 × 5 ml). Chloride (<2%) and Br$^-$ (<500 p.p.m.) remain unchanged. The CCl$_4$ layer is shaken with an aq. soln. containing (NH$_4$)$_2$SO$_4$.Fe$_2$(SO$_4$)$_3$ and Hg(SCN)$_2$ and the I$^-$ are determined photometrically at 460 mμ (Utsumi, *Ibid.*, 1953, **74**, 32). For 0·1 to 6 p.p.m. of I$^-$, the amounts of sample, 18 N H$_2$SO$_4$ and 3% H$_2$O$_2$ used are increased to 50, 2·5 and 5 ml, respectively. There is no interference from common ions.

XVII. Colorimetric determination of a trace of bromide by a catalytic method. Masaru Shiota, Satori Utsumi and Iwaji Iwasaki. *Ibid.*, 1959, **80** (7), 753–757.—The oxidation of I$^-$ to IO$_3^-$ with KMnO$_4$ in H$_2$SO$_4$ is catalysed by Br$^-$(<0·5 p.p.m.); IO$_3^-$ are converted into iodine in the presence of an excess of I$^-$, and the amount of iodine extracted with CCl$_4$ (5 ml) from an H$_2$SO$_4$ soln. (18 N, 0·7 ml per 10 ml of the sample) containing I$^-$ (1 mg per ml, 1 ml) is proportional to the amount of Br$^-$ (0·003 to 0·5 p.p.m.) at a specified temp. (15° or 25°) after a specified lapse of time (210 or 70 sec.). The amount of Cl$^-$ affects the result, which is

corrected with the aid of an empirical table when the amount of Cl$^-$ is known. The iodine in the CCl$_4$ is determined photometrically (Utsumi, *loc. cit.*).   K. SAITO

2222. Some aspects of the polarographic behaviour of technetium and rhenium. R. J. Magee, I. A. P. Scott and C. L. Wilson (Queen's Univ., Belfast, N. Ireland). *Talanta*, 1959, **2** (4), 376–379.—Polarographic studies on soln. of pertechnetate (10^{-5} M in TcO$_4^-$) and perrhenate are reported and discussed. Micro-cells with the dropping mercury electrode, and external calomel and silver electrodes, were used at 25°, and de-aeration was carried out with argon. In 8 N H$_2$SO$_4$ as supporting electrolyte the wave at $E_{\frac{1}{2}} = -0.8$ V can be used for the determination of Re as perrhenate (Re$^{\text{VII}}$→Re$^{\text{IV}}$). In 2 N HCl two waves are obtained for each element, *viz*, $E_{\frac{1}{2}} = -0.65$ V and -1.30 V for Tc, and -1.1 V and -1.7 V for Re. In 2 N NaOH there are well-defined waves at $E_{\frac{1}{2}} = -0.85$ V (for Tc) and -1.1 V (for Re). In 4 N HCl the waves occur at -0.65 V and -0.35 V (for Tc) and -0.1 V and -0.8 V (for Re). In 2 N KOH buffered with Na K tartrate, Tc gives a well-defined wave at $E_{\frac{1}{2}} = -0.65$ V, and this is suitable for the determination of Tc even in low concn.   W. J. BAKER

2223. Direct complexometric determination of iron with xylenol orange as indicator. F. Vydra, R. Přibil and J. Körbl (Chem. Inst., Czech. Acad. Sci., Prague, Czechoslovakia). *Talanta*, 1959, **2** (4), 311–314 (in German).—In the determination of Fe^{3+} by titration at 15° or 60° with 0·05 M EDTA (disodium salt), with xylenol orange as indicator, higher accuracy and a very sharp endpoint are attained by the addition of Fe^{3+} (≃2 g of FeSO$_4$) to the sample soln. (100 ml, pH 1 to 1·5). The usual displacement reaction is thereby replaced by a redox process. Only Bi, Tl^{3+} and large amounts of Cu require preliminary removal; Zr^{4+} and Al (at the higher temp.) should be complexed with NH$_4$F. The soln. should contain ≃60 mg of Fe^{3+}; the standard deviation is ≃±0·07 mg.   W. J. BAKER

2224. Some chel[at]ometric titrations of iron(II). E. Körös and L. Barcza (Inst. of Inorg. and Anal. Chem., L. Eötvös Univ., Budapest, Hungary). *Chemist Analyst*, 1959, **48** (3), 69, 72–73.—Detailed descriptions are given of several modifications of the EDTA titration of Fe in the presence of interfering substances.   G. S. ROBERTS

2225. Determination of iron in phosphate and zirconium salts. F. H. Lohman, D. F. Kuemmel and E. M. Sallee (The Procter & Gamble Co., Cincinnati, Ohio, U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1739–1740.—Iron is separated by extraction into diisopropyl ether from 8 N HCl before photometric determination by means of phenanthroline reagents.   T. R. ANDREW

2226. Elimination of iron from metal solutions by extraction with solvents. R. Rigamonti and E. Spaccamela-Marchetti (Politecnico, Turin, Italy). *Ann. Chim., Roma*, 1959, **49** (1), 106–120.—Attempts to separate Fe from other metals by extraction from a dilute aq. soln. are described. The only successful method is the addition of KSCN and extraction of the Fe(SCN)$_3$ by methyl thiocyanate. Iron, complexed with phenol or salicylic acid, cannot be extracted by amyl alcohol, butanol, butyl acetate, amyl

acetate, amyl formate, *iso*phorone (3:5:5-trimethyl-*cyclo*hex-2-en-1-one) or butyl phthalate from a 0·01% soln. at pH 3·7. Attempts to extract Fe from a soln. containing a 12-molar excess of KSCN by these solvents or by methylammonium acetate, 2-phenoxyethanol, butyl ethyl ketone, or di*iso*butyl ketone gave some extraction but low partition coeff. (amyl alcohol, butanol and the ketones were the best solvents); the best results were obtained with a high SCN to Fe ratio (up to 36) and higher Fe concn. (up to 0·06%); pH (1·2 to 3·7) had little effect. Under these conditions Al, Zn, Ni and Co are extracted to a very much smaller extent. Much better results are obtained by extraction with methyl thiocyanate. The partition coeff. (K) increases with the SCN to Fe ratio up to 3, then remains constant ($K = 47·3$; 97·8% of Fe is extracted), but $K = 6$ without added KSCN. It is shown that $Fe_3(SO_4)_3$ is appreciably sol. in methyl thiocyanate.

CHEM. ABSTR.

2227. Coulometric estimation of ferricyanides. K. S. V. Santhanam and A. P. Madhavan Nair (Alagappa Chettiar Coll. of Technol., Univ. of Madras, India). *Z. anal. Chem.*, 1959, **169** (2), 102–105 (in English).—Determination of $Fe(CN)_6{}^{3-}$ (20 to 50 mg) by controlled-potential coulometry, with a cylindrical silver anode and an inner platinum-gauze cathode (S.C.E. reference electrode), either in H_2O or in Na acetate - acetic acid buffer of pH 5, at $+0·220$ V is accurate to within $±1\%$. With acetate as supporting electrolyte, Cl^- (1 mg) do not interfere.

J. P. STERN

2228. Spectrographic analysis of structural steel by the point-to-plane technique with a copper counter-electrode. P. Murtas (Fiat, Stabilimento Velivoli, Torino). *Metallurg. Ital.*, 1958, **50** (8), 375–378.—Alloying elements (Ni, Cr, Mn, Mo, Si and V) in the range 0·8 to 2·0% are determined by interrupted-spark spectrography. Only a small surface of the specimen is required and graphite electrodes can be replaced by cheaper copper electrodes. In routine analysis the method is satisfactory for amounts >2%.

J. H. WATON

2229. X-ray fluorescence analysis of stainless steel in aqueous solutions. R. W. Jones and R. W. Ashley (Chem. and Metall. Div., Atomic Energy of Canada, Ltd., Chalk River, Ontario, Canada). *Anal. Chem.*, 1959, **31** (10), 1629–1631.—Nickel, Cr and Mo can be determined directly in aq. soln. of stainless steel (prepared by dissolution in aqua regia and treatment by a specified procedure); Nb remains insoluble, and is converted into Nb_2O_5 and determined after briquetting with cellulose powder. The coeff. of variation for all four elements is better than $±1\%$ of the amount present, and the agreement with chemical results on standard samples is within 1%. The method is considerably quicker than chemical techniques and is more accurate and precise than previously reported X-ray methods.

K. A. PROCTOR

2230. Rapid methods for the determination of copper and lead in cast iron and steel. J. Verhun. *Chim. Anal.*, 1959, **41** (8), 332.—(*i*)—Copper in cast iron is determined by dissolving the sample in HNO_3, removing Fe by double pptn. with aq. NH_3, adjusting to pH 2 and electrolysing at 2 amp. for 15 to 20 min. (*ii*)—Copper and Pb in steel are simultaneously determined by dissolving the sample in conc. HCl and adjusting the pH to between 5 and 6 with aq. NH_3 and NH_4Cl. Copper and Pb are then pptd. as sulphides by boiling with $Na_2S_2O_3$, the ppt. is dissolved in HNO_3 and, after dilution of the soln. with water, the analysis is completed by electrolysis at 1 amp. for 15 min. W. T. CARTER

2231. Spectrographic method for the determination of magnesium in spheroidal cast iron. E. Melgara (Lab. Cent., RIV, Torino). *Metallurg. Ital.*, 1958, **50** (8), 378–380.—Experimental conditions are described for determining Mg in spheroidal cast iron in the range 0·01 to 0·15%. J. H. WATON

2232. Photometric determination of aluminium in steel, after separation with ion-exchange resins. M. L. Foglino (Fiat L.R.C.A.A., Torino). *Metallurg. Ital.*, 1958, **50** (8), 372–374.—Aluminium (in the range 0·1 to 2%) is determined spectrophotometrically as its aluminon lake, after removal of interfering elements on an ion-exchange column. *Procedure*—The sample (0·2 g) is dissolved in 15 ml of HCl (1:1) and 15 ml of $HClO_4$, and the soln. is evaporated to fumes. To remove Cr, 10 to 12 drops of conc. HCl are added dropwise; the procedure is repeated 2 or 3 times. The soln. is evaporated to dryness and the residue is dissolved in 15 ml of conc. HCl; this procedure is repeated. The soln. is diluted to 200 ml with 9 M HCl and a 5-ml aliquot is passed through an ion-exchange column (130 mm × 8 mm) of Amberlite IRA-400 (chloride form) at the rate of 1 ml per min. The column is washed with 15 ml of 9 M HCl, and the combined eluates are evaporated to dryness. The residue is dissolved in 0·5 ml of HCl (1:1) and 10 ml of H_2O, and the soln. is transferred to a 50-ml standard flask. Then, in the order given, are added 3 ml of thioglycollic acid soln. (4% in H_2O), 1 ml of pyridine soln. (35% in H_2O) and 15 ml of aluminon buffer soln. (250 g of ammonium acetate dissolved in 500 ml of H_2O, 40 ml of acetic acid, 0·5 g of aluminon dissolved in 25 ml of H_2O and 1 g of benzoic acid in 10 ml of methanol are mixed and diluted to 1 litre). The flask is heated on a boiling-water bath for 5 min., then the soln. is cooled and diluted to volume. The extinction is measured at 546 mμ against a blank. J. H. WATON

2233. Methods for using spectrophotometry in the analysis of steel. I. Determination of aluminium, chromium, phosphorus and silicon. A. Devoti and A. Sommariva (Serv. Chim., Lab. Ric. e Control., Ansaldo S.p.A., Genova). *Metallurg. Ital.*, 1958, **50** (8), 355–366.—The Al is determined with aluminon after the removal of interfering elements by electrolysis at a mercury cathode. The Cr is determined colorimetrically either (*i*) as the Cr^{VI}-diphenylcarbazide complex or (*ii*) directly as the dichromate. In (*i*), V and Mo do not interfere in amounts <5%, whilst (*ii*) is influenced much less by the presence of Ni; high values are obtained in the presence of W, V and Mo. The P is determined as molybdenum blue, which is extracted with *iso*-butyl alcohol after reduction of the molybdophosphate complex with $SnCl_2$. Arsenic and Ge are eliminated as bromides, by addition of NaBr soln. and evaporation with $HClO_4$, and V is extracted with $CHCl_3$ as its diethyldithiocarbamate complex; Cr (≯25%) does not interfere. The Si is determined as molybdenum blue after reduction of the molybdosilicate complex with Fe^{2+}.

J. H. WATON

2234. Spectrophotometric determination of combined carbon in cast iron. M. L. Foglino and G. P. Spagliardi (FIAT L.R.C.A.A., Torino). *Metallurg.*

Ital., 1958, **50** (8), 381–383.—The sample (0·8 g) is treated with 25 ml of acid mixture [H_2O - H_2SO_4 - H_3PO_4 - HNO_3 (10 : 2 : 1 : 7)] on a boiling-water bath. When the reaction slows down, the soln. is heated on asbestos over a Bunsen flame, care being taken to avoid boiling and to replace the water as it evaporates. When the reaction has ceased, the soln. is cooled rapidly and filtered under suction through a sintered glass crucible covered with a thin layer of asbestos. The soln. is diluted to 100 ml and its extinction is measured at 425 mμ. Good agreement is found with values obtained by combustion methods.　　　　J. H. WATON

2235. Amperometric determination of molybdenum in steel by means of fixed electrodes. S. K. Chirkov and L. S. Studenskaya (Lab. of Standard Samples, Ural Inst. of Ferrous Metals). *Zavod. Lab.*, 1959, **25** (9), 1034–1036.—Two similar polarisable platinum-wire electrodes, 0·5 mm in diam., in a soln. of pH between 4 and 6 (acetate buffer) are used for the amperometric titration of Mo with $Pb(NO_3)_2$ soln. Oxidation of Pb^{2+} to PbO_2 occurs on the anode (10 to 12 mm long) and reduction of H^+ occurs on the cathode ($\not< 2$ mm long). The anode potential is 1·10 V *vs.* the S.C.E. The method was tested on samples of ferromolybdenum containing $\simeq 60\%$ of Mo and on samples of steel containing from 0·45 to 5·28% of Mo, with satisfactory results. With steel the sample (1 to 2 g) is dissolved in 30 to 60 ml of HNO_3 (1 : 1) or HCl followed by HNO_3, and the hot soln. is neutralised with 30% NaOH soln. and poured into 50 to 80 ml of hot 30% NaOH soln. and made up to 250 or 500 ml. A filtered aliquot (25 or 50 ml) is mixed with 10 ml of 20% Na acetate soln., then neutralised with dil. HCl or acetic acid to methyl red or litmus, and titrated.　　　　G. S. SMITH

2236. Analytical applications of complexones. XIX. Spectrophotometric and absorptiometric microdetermination of cobalt with 1 : 2-di-(2-aminoethoxy)ethane-NNN′N′-tetra-acetic acid. F. Bermejo Martínez and M. Paz Castro (Univ. of Santiago de Compostela, Spain). *Inf. Quím. Anal.*, 1959, **13** (5), 129–134.—The method is based on measurement of the absorption given by the complex of Co^{3+} with 1 : 2-di-(2-aminoethoxy)ethane-NNN′N′-tetra-acetic acid (disodium salt) (**I**), after oxidation of Co^{2+} with H_2O_2 in ammoniacal soln. in the presence of **I**. The complex is stable, and Beer's law is obeyed within a suitable pH range; 2 μg of Co per ml may be determined spectrophotometrically. The effect of pH on the extinction and the use of some alternative chelating agents (EDTA or 1 : 2-diaminocyclohexane-NNN′N′-tetra-acetic acid) have been examined. Ferric iron, Mn^{2+}, Cu^{2+}, Ni^{2+} and Cr^{3+} interfere.　　　　L. A. O'NEILL

2237. Macro- and micro-determination of traces of cobalt. III. Spectrophotometric determination with nitroso-R salt. W. Haerdi, J. Vogel, D. Monnier and P. E. Wenger (Univ., Genève, Switzerland). *Helv. Chim. Acta*, 1959, **42** (7), 2334–2342 (in French).—A method is described whereby Co in quantities down to 0·01 μg per ml can be determined; Fe^{II} and Cr^{III} interfere seriously, Ni^{II}, Cu^{II} and Fe^{III} only slightly, and Mn^{II}, Zn^{II} and Pb^{II} not at all. Two new types of micro-cell suitable for the Beckman DU instrument are described. Capillary micro-cells with a path length of 1 cm and an internal diam. of 3·5 mm, in conjunction with a photomultiplier, permit the determination of 0·003 μg of Co in 0·3 ml. For quanti-

ties from 0·02 μg to 0·04 μg, macro-cells 1 cm long, capacity 1 to 3 ml, may be used. The test soln. is buffered to pH 6 and heated on a water bath for 1 min. with the reagent (0·05% aq. soln. of nitroso-R salt). A little conc. HNO_3 is added to destroy the excess of reagent, the soln. is centrifuged and the extinction is measured at 520 mμ, at which wavelength the extinction due to the reagent is at a minimum. The error when determining 0·003 μg of Co is $\not>$ $\pm 30\%$.　　　　H. M.

2238. Spectrophotometric determination of cobalt in sodium metal. L. Silverman and R. L. Seitz (Atomics International Div., N. American Aviation, Inc., Canoga Park, Calif.). *U.S. Atomic Energy Comm.*, Rep. NAA-SR-4005, Oct., 1959. 14 pp.—A colorimetric method, with the use of 2-nitroso-1-naphthol, is outlined for determining Co in sodium metal that is used as a coolant in nuclear reactors. The sodium metal is caused to react with water, and the soln. so formed is diluted with HCl, and the cobalt content determined; the cobalt nitrosonaphthoxide is extracted with CCl_4 and the extinction is measured at 535 mμ. The presence of Cl^- is requisite for the extraction of the cobalt complex in dilute soln. Lithium, K and Na have no effect; NH_4^+ have an adverse effect. As little as 0·1 p.p.m. of Co can be determined in sodium metal. To determine Co in the fractional p.p.m. range, 10 g of sample is used The molar extinction coeff. is 11,780.　　　　NUCL. SCI. ABSTR.

2239. Chemico-spectrographic determination of cobalt, copper, tin and zinc impurities in high-purity nickel. D. M. Shvarts and A. I. Granfel'd ("Gipronikel'" Inst.). *Zavod. Lab.*, 1959, **25** (8), 946–948.—The sample of nickel (5 g) is dissolved in 40 ml of HNO_3 (1 : 1), 10 to 12 ml of conc. H_2SO_4 is added, and the soln. is evaporated to fuming. Water (8 to 10 ml) is added, the soln. is evaporated to remove the excess of H_2SO_4, the residue is dissolved in 80 ml of water, the acidity of the soln. is adjusted to between 0·5 and 2 N, and additions of 2 or 3 drops of 0·01 N $KMnO_4$, 10 ml of 40% NH_4SCN and 15 ml of a 2% soln. of diantipyrinylmethane in 0·5 N HCl are made. The soln. is shaken for 30 sec. to give a ppt. of the complexes of Co, Zn, Sn, Cu, Bi, etc. (Ni remains in soln.), then set aside for 30 to 40 min., after which a further 5 ml of diantipyrinylmethane soln. is added, and the soln., after having been shaken again, is extracted with $CHCl_3$ (2 × 10 ml) to dissolve the complexes. The $CHCl_3$ soln. is evaporated to dryness and the contents of metals therein are determined spectrographically.　　　　G. S. SMITH

2240. Spectrographic analysis of nickel I. N. Vitushkina. *Fiz. Sb. L'vov. Univ.*, 1958, [4 (9)], 426–428; *Ref. Zhur., Khim.*, 1959, (21), Abstr. No. 74,677.—The end of a rod-shaped sample (diam. 8 mm) is tapered and washed in HCl; spectra are excited in a low-voltage spark discharge with a 2·5-mm spark gap and a flat-topped carbon electrode; no other details of the circuit are given. A medium spectrograph is used with a 40-sec. burning period and a 10 to 20-sec. exposure. Calibration curves are plotted from the line-pairs—Cu 3273·9 - Ni 3286·9, Fe 2599·3 - Ni 2551·0, and Co 2580·3 - Ni 2551·0 A. By excitation in an a.c. arc, Si is determined from the line-pair Si 2516·1 - Ni 2551·0 A.　　　　K. R. C.

2241. Chel[at]ometric titration of nickel in nickel pellets and nickel - iron alloys. R. Přibil and M. Kopanica (Chem. Inst., Czechoslovak Acad. of Sci.,

Prague). *Chemist Analyst*, 1959, **48** (3), 66–68.—Triethanolamine and EDTA soln. are added to a weakly acid soln. of the nickel and interfering metals. A brown ppt. is formed which, on the addition of NaOH soln., dissolves to give a colourless soln. The Ni - EDTA complex is back-titrated with $CaCl_2$ soln., with the indicator Thymolphthalexone {3:3'-bis-[N N - di(carboxymethyl) aminomethyl] t h y m o l-phthalein}, which is not adversely affected by the Fe^{III} - triethanolamine complex as is Eriochrome black T. The method may be used for samples containing considerable amounts of Fe (and Cu in the presence of thioglycollic acid). Aluminium and some of the Mn are masked, as also are small quantities of Co.   G. S. ROBERTS

2242. Photometric determination of nickel in cobalt and its salts with sodium dimethylglyoxime. W. Nielsch. *Z. Metallk.*, 1959, **50** (4), 234–236.—The neutral test soln. is treated with sufficient KCN to form a complex. The potassium cobaltocyanide formed is oxidised with H_2O_2 to potassium cobalti-cyanide, and the nickel is then extracted by the addition of formaldehyde soln. and Na dimethyl-glyoxime. Finally $CHCl_3$ is added, the soln. is shaken, and the extinction of the nickel dimethyl-glyoxime is measured photometrically at 366 mμ against a blank similarly treated. Nickel, in amounts ranging from 0·001 to 0·5%, can be determined by this method, with only minor varia-tions in the procedure.   NICKEL BULL.

2243. Polarographic determination of impurities in nickel-based alloys by co-precipitation with methyl violet. P. Ya. Yakovlev, G. P. Razumova and R. D. Malinina (Central Sci. Res. Inst. of Ferrous Metal-lurgy). *Zavod. Lab.*, 1959, **25** (9), 1039–1041.—The sample (1 g) is dissolved in 40 to 50 ml of a mixture of HCl and HNO_3 (3 : 1) and the soln. is evaporated to fuming after the addition of 20 ml of conc. H_2SO_4. After dissolution of the salts in 90 to 100 ml of water, the soln. is neutralised with aq. NH_3 and heated to dissolve H_2WO_4. Then 20 ml of 50% tartaric acid soln. and 300 to 350 ml of water are added and the pH is adjusted to between 3 and 4 by treatment with small amounts of HCl. The soln. is mixed with 10 ml of 10% KI soln., 10 ml of 20% NH_4SCN soln. and 30 ml of 1% methyl violet soln. and, after 10 to 15 min., with a further 5 ml of NH_4SCN soln. and a further 20 ml of KI soln. After 10 to 12 hr. the ppt. (containing Zn as thiocyanate and Cd, Pb and Bi as iodides) is collected and washed with a soln. containing 5 or 6 drops of conc. HCl and 10 ml each of the methyl violet, NH_4SCN and KI soln. per litre. The ppt. is ignited at 500° to 550° in the presence of 2 ml of 0·5% $K_2SO_4.Al_2(SO_4)_3.24H_2O$ soln., the ash is dis-solved in 10 ml of conc. HCl, 10 ml of water is added, and Bi and Pb are pptd. by means of aq. NH_3 in the presence of Al as a collector. The ppt. is washed with water containing 2 ml of aq. NH_3 in 500 ml, and dissolved in 15 ml of hot dil. HCl (1:1). The soln. so obtained is evaporated to dryness, the residue is dissolved in 5 ml of conc. HCl, 1 g of Na K tartrate is added, and the pH is brought to 2 with aq. NH_3; 1 ml of 1% gelatin soln. is added, the soln. is diluted to 25 ml, O is removed by a stream of N, and a polarogram is obtained over the range 0 to −0·6 V to give the Bi and Pb. The soln. containing the Cd and Zn is mixed with 10 ml of 20% aq. NH_3, 5 ml of saturated Na_2SO_3 soln. and 1·5 ml of 1% gelatin soln., then diluted to 50 ml, and polarographed over the range −0·5 to −1·3 V.

The determinations are correct to $\simeq$10 to 15% (relative) over the concn. range 0·0010 to 0·0050%.
          G. S. SMITH

2244. Spectrophotometric determination of osmium. II. Sulphanilic acid as a reagent. A. K. Majumdar and J. G. Sen Gupta (Dept. of Inorg. and Anal. Chem., Jadavpur Univ., Calcutta). *Anal. Chim. Acta*, 1959, **21** (3), 260–265 (in English). —Both Os^{VI} and Os^{VIII} form a deep-violet complex with the reagent in the pH range 1·8 to 3·5. The absorption maximum is at 490 mμ and Beer's law is obeyed over the range 1 to 18 p.p.m. for Os^{VI} and 0·5 to 9 p.p.m. for Os^{VIII}. The sensitivities are 0·02 and 0·01 μg per sq. cm, respectively. Many other ions interfere and the Os must be separated by oxidation to H_2OsO_4 with HNO_3 and distillation before determination.   W. T. CARTER

2245. Comparison of precision for solid-, liquid-and powder-sampling techniques in the X-ray fluorescence analysis of high-temperature alloys. S. Friedlander and A. Goldblatt (Chicago Spectro Service Lab., Inc., Ill., U.S.A.). *Appl. Spectro-scopy*, 1959, **13** (4), 91–93.—Coefficients of variation of 0·5% or less are routinely attainable in the determination of major constituents. Absorption and enhancement effects, which are pronounced in solids and briquettes, are completely absent in soln., but the latter have a low sensitivity for minor constituents. A combination of solution and briquetted-oxide techniques provides a method for standardising a solid system for major and minor constituents, as well as a rapid method of analysis for metal samples, regardless of physical form.   K. A. PROCTOR

2246. Spectrographic determination of secondary and trace elements in coal without previous ashing. F. Hegemann, K. Giesen and H. Kostyra. *Ber. dtsch. keram. Ges.*, 1959, **36**, 145–149.—Details are given of the use of a Zeiss Q24 spectrometer with (i) condensed spark excitation for the secondary elements only, and (ii) a d.c. continuous arc for both the secondary and trace elements. The external standard for both procedures was Ni. The results for SiO_2, Al_2O_3, Fe_2O_3, TiO_2, CaO and MgO were checked by chemical analysis. The calibration curves varied considerably from one type of coal to another, but a reproducibility of $\pm2·5$ to $\pm8·5\%$ was achieved.   J. A. SUGDEN

2247. Micro-analysis of silicate and carbonate minerals. III. Determination of silica, phosphoric oxide and metal oxides. J. P. Riley and H. P. Williams (Oceanography Dept., Univ., Liverpool, England). *Mikrochim. Acta*, 1959, (6), 804–824 (in English).—Procedures are detailed for the determination of 10 major-component oxides—SiO_2 (spectrophotometrically with the use of a 5-mg sample after fusion with NaOH); Al_2O_3, total Fe, TiO_2, MnO and P_2O_5 (highly sensitive spectro-photometric methods); CaO and MgO (chelato-metrically by using a photo-electric titrimeter, after removal of interfering elements by extraction with 8-hydroxyquinoline); Na_2O and K_2O (flame-photometrically). (*Cf. Anal Abstr.*, 1960, **7**, 163.)

IV. Determination of aluminium in the presence of interfering elements. J. P. Riley and H. P. Williams. *Ibid.*, 1959, (6), 825–830 (in English).—A comparatively specific spectrophotometric pro-cedure is described for the determination of Al in silicate minerals containing major amounts of interfering elements. Of these, Be, Bi, Cd, Ce, Co,

Cr, Cu, Dy, Fe, Ga, Mo, Ni, Pb, V, W, Y and Zn are removed by extraction with a $CHCl_3$ soln. of 8-hydroxyquinaldine (**I**) at pH 10; Ti can be completely extracted with **I** at pH 4; Al is then extracted with 8-hydroxyquinoline (**II**) at pH 4·5 and the extinction of the yellow soln. is measured at 410 mμ. Since Zr and U are not completely separated from Al by extraction with **I**, the subsequent extraction of Zr with **II** is prevented by complexing it with quinalizarin-3-sulphonic acid, and U is removed by adsorbing it as its anionic acetate complex at pH 4 on a column of Amberlite IRA-400 (acetate form). With samples of $\simeq 25$ mg, satisfactory recoveries of 50 μg of Al were obtained in the presence of at least four times this weight of each of the other elements.   D. F. PHILLIPS

2248. Spectrographic analysis of solutions derived from the decomposition of lead and zinc ores. M. Civera (Centro Ricerche Metallurg., Torino). *Metallurg. Ital.*, 1958, **50** (8), 329–331.—The sample is treated with HNO_3, and to the filtered soln. is added the nitrate of the metal chosen as internal standard (Tl for Zn, Ag for Zn - Fe, and Cu for Fe - Pb). During sparking, the soln. is contained in a small glass cup fitted round the top of the lower graphite electrode, the surface of the soln. covering the top of the electrode with a thin film. In this way it is possible to prevent variations in vol. during sparking, whilst the limited area of electrode exposed to the soln. permits the use of the electrode for a large number of experiments. Procedures are given for the spectrographic determination of Zn, Zn and Fe together, and Fe and Pb together.   J. H. WATON

2249. Spectrographic determination of certain oxides in cement. L. S. Likhodel and N. I. Nosenko. *Fiz. Sb. L'vov. Univ.*, 1958, [4(9)], 471–474; *Ref. Zhur., Khim.*, 1959, (21), Abstr. No. 74,679.—The oxides studied are Al_2O_3, Fe_2O_3, SiO_2, TiO_2, MgO and CaO. The cement is powdered and mixed with CuO and powdered charcoal in the ratio of 1:1:5; the mixture (100 mg) is moistened with ethanol and spread in a thin layer on a copper plate. Spectra are excited in a 5-amp. a.c. arc with a conical copper upper electrode and a 60 to 65-sec. exposure. The copper plate is moved relatively to the upper electrode with a 2·5-mm electrode gap. Synthetic standards are prepared from oxides which are then fired at 1600° for 2 hr. Calibration curves are plotted for $\log(I_x/I_{Cu})$ *vs.* $\log C$ for the line-pairs (in A): Ca 3344·5 - Cu 3063·4; Si 2435·1 - Cu 2723·9; Mg 3096·8 - Cu 3063·4; Fe 2395·6 - Cu 2441·6; Al 2367·1 - Cu 2369·9; and Ti 3088·2 - Cu 3073·8. Results from chemical determinations are in good agreement with spectrographic results. The test takes $\simeq 2$ hr.   K. R. C.

2250. Determination of chlorides and fluorides in glass of high boron content. F. Plesnivý (Res. Inst. Vacuum Electrotech., Prague). *Sklář a Keram.*, 1959, **9** (7), 214–215.—Two 0·25-g samples are fused with Na_2CO_3 and the melts are dissolved in water. One soln. is just acidified with HNO_3, treated with HNO_3 - H_2SO_4 and 0·5% $AgNO_3$ soln. and made up to 50 ml, and the turbidity is measured after 15 min. at 420 mμ. Results are referred to a calibration curve (0·025 to 0·35 mg of Cl⁻ in 50 ml). The second soln. is acidified with H_3PO_4, and the F⁻ are distilled at 190° $\pm$ 5° (as H_2SiF_6) into Na_2CO_3 soln. The distillate is evaporated to dryness and the residue is fused; the melt is dissolved in water and neutralised with HCl, and the silicic acid is

filtered off. The filtrate is made up to 100 ml, an aliquot is treated with a soln. of zirconium - alizarin lake and made up to 50 ml, and the extinction is read after 10 min. at 520 mμ. Results are referred to a calibration curve (0·01 to 0·1 mg of F⁻ in 50 ml).   J. ZÝKA

See also Abstracts—**2032**, Recent developments in determination of metals; carbon in steel. **2033**. Inorganic micro-analysis. **2037**, Substituted benzidines as indicators for titrations of $Cr_2O_7^{2-}$ and Ce^{4+}. **2040**, **2041**, Siloxene as indicator. **2042**, **2043**, Indicators for complexometric titrations. **2263**, Argon in gas mixtures. **2458**, Radionuclides in milk ash. **2495**, Sulphides in water. **2496**, Boron in water. **2498**, Iron and Al in sea water. **2499**, Sulphate in super-phosphate. **2528**, Separation of Cu, Cd and Bi. **2567**, Fluorescence X-ray spectrography for Mn, Fe and Ca in ores. **2570**, Flame photometry of Rb and K. **2575**, Gases in metals.

3.—ORGANIC ANALYSIS

Determination of elements and radicals and of organic compounds not included in other sections. Organic industrial products, including petroleum and its products, fuels, detergents, volatile oils, cosmetics, dyestuffs, fibres, plastics, resins, paints, elastomers, leather, explosives.

2251. Sealed-tube combustions for the determination of carbon-14 and total carbon. D. L. Buchanan and B. J. Corcoran (Radio-isotope Service, Veterans Admin. Hospital, West Haven, Conn., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1635–1638.—The method is a development of that of Wilzbach and Sykes (*cf. Anal. Abstr.*, 1955, **2**, 91) and avoids the formation of oxides of nitrogen, which interfere with the performance of a proportional counter, from samples that contain nitrogen. *Procedure*—Add a mixture of CuO (previously heated to 850° and cooled), MnO_2 and $CuCl_2$ (5:1:1) (1 g) to the sample (5 to 10 mg) contained in a Vycor 7900 combustion tube. Evacuate and seal the tube, mix the contents and heat in a muffle-furnace at 850° for 30 min. Cool and open in a vacuum system similar to that described previously (*cf.* Buchanan and Nakao, *J. Amer. Chem. Soc.*, 1952, **74**, 2389), measure the total vol. of CO_2, and determine the amount of ^{14}C in a proportional gas counter. The determination is as accurate and precise as by other methods, and the CO_2 is unusually pure.   A. R. ROGERS

2252. Isotope distribution in the Unterzaucher analysis of labelled oxygen compounds. W. G. Miller and L. Anderson (Dept. of Chem., Coll. of Letters and Sciences, Univ. of Wisconsin, Madison, U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1668–1669.—The internal distribution of oxygen isotopes in CO_2 obtained from enriched water by the Unterzaucher method is not random. This finding is important in the calculation of the ^{18}O content of samples which have been converted into CO_2 by the Unterzaucher method preparatory to mass-spectrometric analysis. A simple equation for making these calculations is given.   A. R. ROGERS
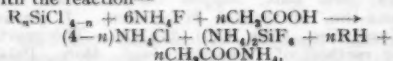
2253. Modification of the Grote - Krekeler apparatus. M. Přibyl and J. Havíř (Military Acad., Brno, Czechoslovakia). *Chem. Listy*, 1959, **53** (8), 856–857.—The absorption part of the Grote - Krekeler

apparatus (*cf. Angew. Chem.*, 1933, **46**, 106) was replaced by two absorption vessels which are connected with the combustion tube. A Mariotte flask is placed behind the second absorption vessel instead of the pump. The modified apparatus was used successfully in the routine analysis of chlorinated products (0·1 to 0·4 g of sample) containing 1 to 70% of chlorine. J. ZÝKA

2254. Automation of the Pregl nitrogen determination. G. Kainz and L. Hainberger (Univ. Wien, Austria). *Z. anal. Chem.*, 1959, **169** (1), 27-34 (in German).—Details are given of an automatic apparatus for the determination of N by Pregl's method. The combustion is regulated by a flowmeter placed in front of the combustion tube, which actuates a relay that controls the burners. The rate of flow in the combustion zone is always kept constant. Traces of N in petroleum can be accurately determined in a sample of 0·1 g. S. M. MARSH

2255. Rapid micro-determination of halogens in organic compounds. N. V. Sokolova, V. A. Orestova and N. A. Nikolaeva (Inst. of High-molecular Compounds, Acad. Sci. USSR, Leningrad). *Zhur. Anal. Khim.*, 1959, **14** (4), 472-477.—The sample (usually 3 to 15 mg, or ≯ 50 mg) is wrapped in an ashless filter-paper (or put in a capillary if a liquid), which is held in a piece of platinum gauze (20 mm × 30 mm) attached via a platinum wire to the ground glass stopper of a 200 to 350-ml conical flask having a side arm with a tap, and containing 10 ml of water and 1 ml of 2 N KOH (for determining iodine) and 3 drops of H_2O_2 (when determining Cl and Br). The air in the flask is replaced with oxygen, and the filter-paper set alight. After the burning, the flask is vigorously shaken for 6 to 10 min., the tap is opened to let in air, and the stopper, platinum wire and gauze are washed down; the contents of the flask are boiled for 5 min. to destroy H_2O_2, then cooled, and acidified to a pH of 2·3 with 0·5 N HNO_3. The chloride or bromide is titrated with 0·01 N mercuric nitrate or perchlorate to a pale pink colour, with diphenylcarbazone as indicator. Iodine is determined by oxidation to iodate and titration with 0·02 N $Na_2S_2O_3$, with starch as indicator. The method has been tested on a large number of organic halogen compounds; the accuracy is ±0·3%. C. D. KOPKIN

2256. Titrimetric determination of chlorine in organosilanes. Ya. I. Chulkov. USSR Pat. No. 118,224; 20th Feb., 1959.—The silanes are decomposed with NH_4F in glacial acetic acid in accordance with the reaction—

$$R_nSiCl_{4-n} + 6NH_4F + nCH_3COOH \longrightarrow$$
$$(4-n)NH_4Cl + (NH_4)_2SiF_6 + nRH +$$
$$nCH_3COONH_4,$$

and Cl^- are determined by known methods. In the example given, 5 ml of glacial acetic acid, 2·5 ml of acetic anhydride and 4 g of NH_4F are added to a dry 100-ml conical flask, a sealed ampoule containing the sample (0·2 to 0·6 g) is added, and the ampoule is broken. A crystalline ppt. forms, and is dissolved by adding 3 ml of HNO_3 (sp. gr. 1·4) and 10 to 15 ml of water. The clear soln. is made up to 100 ml, mixed, and an aliquot is titrated to determine Cl^-. The mercurimetric titration, with sodium nitroprusside as indicator, is described. Carbonates, acetates and borates do not interfere in this titration; large amounts of ammonium salts interfere. In determining Cl^- in

CH_3SiCl_3, $(CH_3)_4SiCl_2$, $(C_2H_5)_2SiCl_2$, $(C_6H_5)(CH_3)SiCl_2$ and $C_6H_5SiCl_3$ the deviations from the calculated values were from −0·07 to +0·18%.
C. D. KOPKIN

2257. Semi-micro determination of silicon and phosphorus in organic compounds. T. R. F. W. Fennell and J. R. Webb (R.A.E., Farnborough, Hants, England). *Talanta*, 1959, **2** (4), 389-391.—Two alternative methods for determining Si and P in a single sample of a liquid of high boiling-point are given. (*i*) The sample is digested with HNO_3 and H_2SO_4 in a glass tube, the liquid is decanted, and the P therein is determined gravimetrically by pptn. with Jorgensen's reagent [nitratopentammino-cobaltinitrite (*cf.* Belcher and Godbert, *Analyst*, 1941, **66**, 194)]. The residue of SiO_2 is washed with HNO_3 (1:1) and then with ethanol, dried at 100°, weighed, ignited and weighed again. The content of Si is calculated from the corrected value for SiO_2. (*ii*) The sample is decomposed with Na_2O_2 in a micro-bomb, the acidified leachings are evaporated to dryness and, after baking at 110°, the residue is extracted with hot water. The soln. is filtered and P is determined gravimetrically in the filtrate as quinoline molybdophosphate. The SiO_2 in the insol. residue is determined by treatment with HF. The error is within ±0·3% for both methods, but (*ii*) is preferred. W. J. BAKER

2258. Identification of alkyl(aryl)mercury radicals and quantitative determination of mercury in N-organomercury compounds. B. Hetnarski and K. Hetnarska (Lab. of Organic Synthesis, Polish Acad. Sci., Warsaw). *Bull. Acad. Polon. Sci., Sér. Chim. Géol. et Géogr.*, 1959, **7** (9), 645-650 (in English).—For the identification of the organomercury radical, an ethanolic soln. of the sample is heated to boiling for ≃5 min. with the stoicheiometric amount of a thiol, *e.g.*, 2-mercaptobenzo-thiazole (**I**); the stoicheiometric amount of sodium ethoxide in abs. ethanol is then added and, after the reaction is complete, the mixture is diluted with H_2O to precipitate the almost insol. cryst. S-organomercury compound. This is collected and its m.p. determined; m.p. of derivatives from 15 compounds are listed. The content of Hg can be determined, to within ±0·3%, by heating ≃0·1 g of the sample to boiling with 25 ml of a 0·01 N soln. of the Na salt of **I** in ethanol and then adding 200 ml of a saturated aq. soln. of NaCl to precipitate the S-organomercury deriv. This is filtered off and the excess of **I** in the filtrate is titrated with 0·01 N iodine in the presence of 1% Na acetate soln. (10 ml). W. J. BAKER

2259. Information on the anomalous reactions in the amino-nitrogen determination. III. Anomaly of compounds with active methylene groups. G. Kainz, F. Kasler and H. Huber (II Chem. Inst., Univ., Vienna, Austria). *Mikrochim. Acta*, 1959, (6), 875-882 (in German).—Attempts are made to explain the reactions between HNO_2 and compounds containing an active methylene group, which give rise to errors in the van Slyke amino-N determination. Details of the reaction of HNO_2 with malonic acid, malonic ester, ethyl acetoacetate and acetylacetone are given.

IV. Anomalous reaction of glycine in the amino-nitrogen determination of van Slyke. G. Kainz, F. Kasler and H. Huber. *Ibid.*, 1959, (6), 883-889 (in German).—The reaction scheme of Austin (*J. Chem. Soc.*, 1950, 149) does not adequately explain the anomalous amounts of gas obtained in

the reaction of glycine with HNO_2. Following a re-investigation, new explanations of the competing reactions involved are offered.

V. The anomaly of phenols. G. Kainz and H. Huber. *Ibid.*, 1959, (6), 891–902 (in German).— Explanations are offered for the reactions of phenols with HNO_2 which lead to the anomalous gas volumes (of N and N_2O) that occur in the van Slyke determination.

VI. The anomaly of indole and its derivatives. G. Kainz and H. Huber. *Ibid.*, 1959, (6), 903–907 (in German).—An explanation, based on chemical preparations, is offered for the high gas volumes delivered, during the amino-N determination, by indole and many of its derivatives, even although these compounds contain no primary amino group.

D. F. Phillips

2260. Quantitative determination of methylene groups in open-chain compounds containing at least four such groups. E. A. Glebovskaya, É. I. Maksimov and A. K. Petrov (All-Union Sci.-Res. Inst. of Petroleum Geology and Prospecting, Leningrad). *Zhur. Anal. Khim.*, 1959, **14** (4), 478–482.—This determination may be carried out by measuring the integral absorption in the region 13·2 to 14·4 μ, *i.e.*, the area bounded by the extinction curve and its base line. For solids and liquids the number of methylene groups, n, is proportional to SV_M, where S is the area (sq. cm) under the curve and V_M is the mol. vol. (approx. equal to the mol. wt. divided by the density). Also, the value $K = S/cdx$ is constant, where c is the percentage (by wt.) of methylene groups, d is the specific gravity of the substance, and x is the thickness (cm) of the layer. For substances in solution the percentage (by wt.) of methylene groups can be calculated without knowing the density. The accuracy of determining methylene groups is ± 1 group or $\simeq 10\%$ (by wt.) of such groups. It is necessary to determine the proportionality constants with pure substances, since they vary with the instrument and experimental conditions used.

C. D. Kopkin

2261. Oxidimetric hydroxylamine method of carbonyl determination. B. Budĕšínský and J. Körbl (Forschungsinst. für Pharmazie und Biochem., Prague). *Mikrochim. Acta*, 1959, (6), 922–931 (in German).—The method is based on the reaction of the carbonyl group with excess of hydroxylamine, followed by titration of the unchanged hydroxylamine with $0·1 M K_3Fe(CN)_6$ or with $0·1 N$ iodine, the former being preferred. The titration is carried out in strong alkaline soln. with potentiometric measurement of the end-point. Under the conditions stated, the reaction may be completed within 1 hr. Results are given for 26 carbonyl-containing compounds.

D. F. Phillips

2262. Determination of ethoxyl groups in certain organosilicon and organoaluminium compounds. E. A. Bondarevskaya, S. V. Syavtsillo and R. N. Potsepkina. *Zhur. Anal. Khim.*, 1959, **14** (4), 501–503.—The method is based on hydrolysis of the compounds with the formation of ethanol, which is determined by known methods. For organosilicon compounds, place a sealed ampoule containing the sample (0·12 to 0·15 g) in a flask containing 10 ml of 5% $K_2Cr_2O_7$ soln. and 5 ml of H_2SO_4 (1:1), break the ampoule under the liquid surface, heat the flask under reflux on a boiling-water bath for 30 min., cool, add 25 ml of 10% KI soln. and after

5 min. titrate the liberated iodine with $0·1 N$ $Na_2S_2O_3$. Carry out a blank simultaneously. The method can be used to determine 0·1% of ethoxyl groups; the maximum relative error obtained in a number of analyses was 12%. The method may be adapted for the determination of ethoxyl groups in organoaluminium compounds by working in an atmosphere of N.

C. D. Kopkin

2263. Determination of argon and methane in gas mixtures—accuracy and precision of analysis using a simple gas-chromatographic apparatus. J. Lacy and R. V. Hill (African Explosives and Chem. Ind. Ltd., P.O. Northrand, Transvaal). *Chem. & Ind.*, 1959, (37), 1148–1149.—In the technique described the column used is of glass tubing (6 ft. long and 6 mm internal diam.) bent in the form of a U and packed with activated carbon. A carrier gas flow-rate of 50 ml of N per min. was maintained throughout. A differential thermal-conductivity type detector was used, each of the two cells consisting of 9·5 cm of 42-gauge platinum wire working with a bridge circuit in conjunction with a 0 to 10-mV Speedomax pen recorder. Results for synthetic gas mixtures show coeff. of variation of $< 0·2\%$. A simple apparatus for the preparation of these gas mixtures is illustrated.

E. G. Cummins

2264. Gas-chromatography analysis of the reaction products from the hydroformylation of isobutene. G. W. Warren, J. F. Haskin, R. E. Kourey and V. A. Yarborough (Development Dept., Union Carbide Chemicals Co., South Charleston, W. Va., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1624–1626.—The procedure described is more rapid than, but as accurate and precise as, alternative chemical and instrumental techniques. Some 20 compounds, generally in concn. $> 0·1\%$ and boiling between 20° and 176°, are present in the sample and most of the major components can be resolved. The chromatography is carried out on a column (4 metres $\times$ 6·3 mm) of 30% (w/w) of N-phenyl-1-naphthylamine on 100 to 120-mesh Celite, at 100°, with a helium gas flow of 100 ml per min. Quant. measurements were made by the area percentage method, and for one sample examined six times consecutively the limits of uncertainty at the 95% confidence level ranged from 3 to 4% for major constituents to 100% for traces (0·1%).

K. A. Proctor

2265. Determination of alkylketen dimers with ethylamine. Ichiro Imai, Takeo Wakabayashi, Machio Yoshino, Shigeru Komiya and Kyoko Kotera (Res. Lab., Nippon Oil and Fats Co., Oohama-cho, Amagasaki, Hyogo-ken). *J. Japan Oil Chem. Soc.*, 1959, **8** (7), 279–282.—The gravimetric method of Piekarski (*Anal. Abstr.*, 1954, **1**, 1880), involving the use of the amine adduct of alkylketen dimers (**I**), was modified so as to titrate the excess of ethylamine (**II**) with HCl. Diethyl ether and ethanol are the best solvents for the sample and the reagent, respectively; ethanol reacts with **I** much more slowly than does **II**. The results obtained by this method and the iodine-value method agree well for homologues higher than the decyl dimer, but those obtained by the iodine-value method are lower for homologues lower than the octyl dimer. *Procedure*—Dissolve the sample (0·2 to 1 g) in ether (30 ml), add $0·1 N$ **II** in ethanol (20 ml) and titrate with $0·1 N$ HCl (bromocresol green).

K. Saito

2266. Determination of methanol in ethanol with Schiff's reagent. O. Oja, R. J. Peltonen and M. Koski (State Alcohol Monopoly, Helsinki, Finland). *Z. anal. Chem.*, 1959, **169** (5), 321–327 (in German).—Statistical calculations from a large number of results indicate that erratic results may be obtained by the use of a freshly prepared Schiff's reagent. The dye Fuchsin Diamant (large crystals) No. 1358 (Merck) was superior to Diamantfuchsin I, No. 1356 (Merck).   N. E.

2267. Polarographic determination of methanol and ethanol. J. Kubis (Chem. Inst., Med. Fac. Komenský Univ., Bratislava, Czechoslovakia). *Pracovní Lékařství*, 1959, **11** (9), 465–468.—Both alcohols are distilled through a layer of granulated fused PbCrO$_4$ at 300° (3 ml per 15 min.); 3·4% of the methanol is oxidised to formaldehyde and 15·9% of the ethanol to acetaldehyde. The aldehydes are then determined polarographically in 0·1 N LiCl as supporting electrolyte (two separate waves). The determination of 5 mg of methanol and 1 mg of ethanol per 100 ml is complete within 1 hr.   J. ZÝKA

2268. Gas-chromatographic analysis of crude methanol and higher alcohols. Tamechika Yamamoto and Kazu Saito (Japan Gas Chem. Ind., Enoki-cho, Niigata). *J. Soc. Org. Synth. Chem. Japan*, 1959, **17** (5), 293–301.—For the analysis of the crude methanol produced from H and CO obtained from natural gas, gas chromatography with octadecyl alcohol (25 g) on Celite No. 408 (Johns Manville) (100 g, length of column 70 cm) as the solid phase and He as the carrier gas is satisfactory at 60°. Besides methanol (84%) and water (14%), dimethyl and diethyl ethers (1·2%), methyl formate and aliphatic alcohols (C$_2$ to C$_7$) were determined with a relative error of $\simeq 3\%$.   K. SAITO

2269. New data for the densitometric determination of methanol in formalin. A. A. Skelding and R. F. Ashbolt (Synthite Ltd., Ryders Green, West Bromwich, England). *Chem. & Ind.*, 1959, (39), 1204–1207.—The sp. gr. of accurately prepared soln. of six different mixtures of aq. formaldehyde and methanol were measured by pyknometer and by a glass thermometer plummet. Formaldehyde was determined by use of alkaline H$_2$O$_2$ and methanol, after distillation with alkaline H$_2$O$_2$, by Walker's method (*cf.* "Formaldehyde," Reinhold, N.Y., 1953, 2nd Ed., p. 395). A table of sp. gr. at 15°/15° and 25°/25° *vs.* methanol (%) and formaldehyde (%) has been prepared, of which a part, covering commercial formalin, is reproduced. The errors of other published tables are discussed.
                E. J. H. BIRCH

2270. Determination of isopropyl alcohol in presence of other alcohols. H. Wehle (Hygiene-Inst., Univ., Leipzig, Germany). *Z. anal. Chem.*, 1959, **169** (4), 241–247 (in German).—*iso*Propyl alcohol (I) differs from methanol, ethanol and *n*-propanol in being oxidised by Br in conc. aq. acid, or in satd. aq. KHSO$_4$; the acetone produced is further brominated, but the Br taken up in the further reaction can be determined iodimetrically. By difference, the amount of I can be quant. determined with an accuracy within $\pm 3\%$; the other alcohols named and acetone do not interfere. In the analysis of cosmetics, volatile organic oils must be removed by preliminary extraction with pentane. *Procedure*—Keep the sample (5 to 15 ml containing 0·2 to 0·3% of alcohol) with 0·1 N

KBr - KBrO$_3$ (25 ml) and conc. H$_3$PO$_4$ (10 ml) in a sealed flask at 40° for 1 hr. Cool, add rapidly 20% KI soln. (10 ml) and keep (sealed) at 90° for 15 min. Cool, titrate with 0·1 N Na$_2$S$_2$O$_3$, and compare with a water blank.   J. P. STERN

2271. Analysis of 1:2-glycols and polyhydric compounds. II. Iodimetric determination of tartaric acid in the presence of citric acid via the glyoxylic acid formed by periodate oxidation. E. Schulek and L. Maros (Inst. for Inorg. and Anal. Chem., L. Eötvös Sci. Univ., Budapest). *Magyar Kém. Foly.*, 1959, **65** (9), 363–365 (in Hungarian); *Acta Chim. Acad. Sci. Hung.*, 1959, **20** (4), 443–449 (in German).—Tartaric acid (I) is oxidised to glyoxylic acid (II) with HIO$_4$; II is converted into its bisulphite derivative with the simultaneous reduction of all oxidising agents present. After the removal of the excess of NaHSO$_3$ the bisulphite derivative of II is decomposed with CN$^-$ to give SO$_3^{2-}$ which are determined iodimetrically. *Procedure*—Into a 100-ml flask measure 10 ml of a soln. containing I (1·8 to 2 g) in H$_2$O (500 ml). Add 20% H$_3$PO$_4$ soln. (2 ml), followed by 96% ethanol (10 ml) and 0·1 M HIO$_4$ (5 ml). After 30 min. add 1·5 M NaHSO$_3$ until the colour of iodine is discharged, and 0·5 ml in excess. Neutralise with 20% NaOH soln. in the presence of methyl red soln. (1 ml) and acidify with 10% acetic acid soln. (1 drop). Cool if necessary, and shake the flask; remove the stopper at intervals. Add pentane (5 ml) and after 30 min. add starch soln. (0·2%, 10 drops) and oxidise most of the excess of HSO$_3^-$ with 5% iodine soln. Shake the flask vigorously near the brownish-violet end-point and continue the titration with 0·1 N iodine. Add 20% NaOH soln. (3 ml) and solid KCN (0·2 to 0·3 g) and mix gently, without breaking the pentane layer. After 3 to 4 min. neutralise the soln. with 20% HCl soln. and add 1 drop in excess. Titrate the soln., with slow mixing, with 0·1 N iodine; shake the flask near the end-point. The equiv. wt. of I is one-fourth of its mol. wt. Hydroxy acids not containing a vicinal diol group do not interfere (*e.g.*, the accuracy is good even in the presence of a 50 to 100-fold excess of citric acid).   A. G. PETO

2272. Distinguishing xylose from arabinose and other sugars. B. Drożdż (Pharmacognosy Dept., Poznan). *Acta Biochim. Polon.*, 1959, **6** (3), 369–372.—A rapid identification is best obtained by drop analysis on Whatman No. 1 chromatographic paper. The sugar soln. (1 to 10 μg) is applied to the paper which, after being dried, is sprayed with a 1% soln. of barbituric acid (in glacial acetic acid) or a 0·05% soln. of thiobarbituric acid (in glacial acetic acid) and then dried at 125° (10 to 15 min.). Yellow spots appear which, when moistened over boiling water, take on a colour characteristic of the sugar present, namely xylose, dark green; arabinose, pale brown.   B. LAKE

2273. Refractive indices of pure sucrose solutions. D. F. Charles (Calif. and Hawaiian Sugar Refining Corp. Ltd., Crockett, Calif., U.S.A.). *Int. Sugar J.*, 1959, **61**, 236–240.—The refractive indices of sucrose soln. in the concn. range of 50% to 85% have been precisely determined. The results confirm that the International Sugar Scale (1936) is in error by 0·08% at a concn. of 66% of solids and by 0·20% at a concn. of 80%. The test soln. were made by weighing pure sucrose and water. Corrections were made for adsorbed and occluded moisture. The concn. of the soln. was checked by pyknometer

measurements. Tests for reducing substances showed that decomposition was negligible. The refractive-index measurements were made with a Bausch and Lomb precision refractometer.

G. Burger

2274. Separation of sugars on ion-exchange resins. J. K. N. Jones, R. A. Wall and A. O. Pittet (Gordon Hall, Queen's Univ., Kingston, Ontario, Canada). *Chem. & Ind.*, 1959, (38), 1196.—A mixture of sucrose, raffinose and glucose (200 mg of each) can be separated into chromatographically pure components by a 24-hr. elution with water from a column (100 cm × 2 cm) of Dowex 50W (2% DVB, 200 to 400 mesh, Li+ form). With simple sugars elution always proceeds in the order of descending mol. wt., but with methylated sugars this order is reversed, the most highly methylated fractions eluting last. Hydrolysates of methylated cherry-gum and methylated starch can be separated into fractions according to the degree of methylation. 2:3:5-Tri-O-methyl-L-arabinose was isolated from the methylated gum and 2:3:4:6-tetra-O-methyl-D-glucose from the methylated starch.

W. J. Baker

2275. Uniform methods of analysis for starch and starch hydrolysis products. K. Heyns (Chem. Staatsinst., Hamburg). *Stärke*, 1959, **11** (1), 68–74.—The method for dextrose and maltose of Patterson and Savage (*Analyst*, 1957, **82**, 812) is recommended.

N. E.

2276. 2:4-Dinitrophenylhydrazine, a suitable reagent for the colorimetric determination of carbonyl compounds. M. Pesez (Res. Lab., Roussel-Uclaf, Paris). *J. Pharm. Pharmacol.*, 1959, **11** (8), 475–476.—Dissolve the sample (10 to 100 μg) in acetic acid (1 ml) and add a 0·1% soln. of 2:4-dinitrophenyl-hydrazine in acetic acid containing 0·5% of conc. HCl (5 ml). Set aside in the dark for 1 hr. at room temperature and measure the extinction at 412·5 mμ. The reagent blank is negligibly small. For benzo-phenone, camphor and sugars, heat the mixture for 15 min. on a boiling-water bath and measure at 432·5 mμ. For steroids, heat for 15 min., cool to 20°, add a M soln. of K acetate in ethanol (0·5 ml) and measure at 412·5 mμ. Of about 40 carbonyl compounds tested, only 11-oxosteroids did not respond to the test.

A. R. Rogers

2277. Separation and micro-determination of formaldehyde and methanol in an excess of azo-alkanes, methylal and lower esters. F. Wenger and K. O. Kutschke (Div. of Pure Chem., Nat. Res. Lab., Ottawa, Canada). *Anal. Chim. Acta*, 1959, **21** (3), 296 (in English).—Azomethane can be removed quantitatively from aq. soln. containing formaldehyde and methanol by blowing nitrogen through the soln. at room temperature. Samples containing up to 60 p.p.m. of formaldehyde and 220 p.p.m. of methanol can be treated without significant loss of either component. After the removal of the azomethane, methanol is separated from the formaldehyde by distillation in the presence of chromotropic acid and, after oxidation, is determined as formaldehyde (*cf.* Boos, *Anal. Chem.*, 1948, **20**, 964). The formaldehyde content of the residue is determined by the Bricker and Vail method (*cf. Brit. Abstr. C*, 1950, 361).

W. T. Carter

2278. Rapid determination of acetaldehyde in ethylene oxide vapour with a detector tube. Yoshitaka Kobayashi and Yasuzo Murata (Fac. of Engng,

Yokohama Univ., Minami-ku, Yokohama). *J. Soc. Org. Synth. Chem. Japan*, 1959, **17** (5), 283–286.—Silica gel containing hydroxyammonium sulphate (**I**) and thymol blue (**II**) is the best absorbing agent for the determination of acetaldehyde (**III**) (0·003 to 0·3%) in ethylene oxide. Both the rate of flow and the temp. affect the result, which is corrected by means of empirical diagrams. Other aldehydes, ketones, acid gases (*e.g.*, HCl and SO$_2$) and oxidising gases (*e.g.*, Cl, ClO$_2$ and NO$_2$) interfere. Mix silica gel dried in air at 110° (40 to 60 mesh, 100 g) with **I** soln. (1%, 90 ml) and **II** (0·1% in ethanol, 20 ml) and dry *in vacuo* until the water content is 20%. Place the product (0·15 g) in a thin glass tube (diameter, 2 mm). Pass the sample gas (30 to 100 ml) through the tube at 0·5 ml per sec. and determine the **III** content by means of empirical tables.

K. Saito

2279. Chromatography of mixtures of lactalde-hyde, acetol [hydroxypropan-2-one] and pyruvalde-hyde on bisulphite ion-exchange columns. E. Huff (Lab. of Clin. Invest., Nat. Inst. of Allergy and Infectious Diseases, Bethesda, Md., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1626–1629.—Adsorb the sample of carbonyl compounds (<200 μmoles) from aq. soln. (10 to 100 ml) on to a column (60 cm × 1 cm) of Dowex 1-X10 (SO$_3$H− form) and elute in turn with 0·1 M, 0·2 M, 0·4 M, 0·8 M and 1·6 M potassium bisulphite soln. (90 ml of each). Add the eluents in 3-ml portions; allow each portion to drain under gravity and collect and analyse separately. For fractions that are 0·1 M or 0·2 M with respect to bisulphite, add 0·8 M bisulphite to make the final concn. 0·4 M and allow to react at 24° for 1 hr. For fractions 0·4 M, 0·8 M or 1·6 M with respect to bisulphite, add aq. HCl and 1·6 M bisulphite to give a concn. of 0·8 M and a pH of 2·5, and allow to react at 24° for 2 hr. Add ice-cold H$_2$O (20 ml), stir by passage of air, add a slight excess of N iodine at 0° and titrate in succession with 0·1 N Na$_2$S$_2$O$_3$, 0·01 N iodine, 0·01 N Na$_2$S$_2$O$_3$ and 0·005 N iodine (all at 0°), with starch as indicator. Add an excess of NaHCO$_3$ to liberate the bound sulphite and titrate rapidly with 0·005 N iodine. A procedure is also given for the recovery of the carbonyl compounds from the pooled eluates.

A. R. Rogers

2280. High-voltage paper electrophoresis of organic acids and determination of migration rates. D. Gross (Tate and Lyle Res. Lab., Keston, Kent, England). *Chem. & Ind.*, 1959, (39), 1219–1220.—The migration of 54 non-volatile organic acids (43 aliphatic, 11 aromatic) in an enclosed-strip type apparatus, with aluminium cooling plates, polyethylene insulation and pneumatic pressure control, at pH 2, 4 and 8·9, is determined in comparison with the migration of the chloride ion (absolute migration—37·1 × 10^{-5} cm per sec. per V at pH 2; 36·6 × 10^{-5} at pH 4, and 29·0 × 10^{-5} at pH 8·9 at the appropriate temp.). The conditions used were—at pH 2·0 (0·75 M formic acid), 100 V per cm, 6·5 mA per cm, 25 min., temp of cooling water, 15·5°; at pH 4·0 (0·5 M acetic acid adjusted with pyridine), 100 V per cm, 9·6 mA per cm, 20 min., temp., 10°; and at pH 8·9 (0·1 M ammonium carbonate), 80 V per cm, 8 mA per cm, 25 min., temp., 8°. Sucrose was used as a non-migrating marker.

E. J. H. Birch

2281. Colorimetric estimation of citric acid. R. E. Macdonald and W. E. Waterbury (Lab. of Bacteriology, Cornell Univ., Ithaca, N.Y., U.S.A.). *Nature*, 1959, **184** (Suppl. 13), 988–989.—The

method described is a modification of the combined methods of Natelson *et al.* (*J. Biol. Chem.*, 1948, **175**, 745) and Cartier and Pin (*Bull. Soc. Chim. Biol.*, 1949, **31**, 1176). The sensitivity of the method, which is easy to perform and not subject to day-to-day variation, is about ±3%. Hydrazine sulphate is used to decolorise the $KMnO_4$ instead of H_2O_2, and this may account for the better over-all stability of the method.

K. A. PROCTOR

2282. Chromatographic analysis of mixtures of adipic, glutaric and succinic acids. A. I. Smith (The Chemstrand Corp., Decatur, Ala., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1621–1624.—Mix together silicic acid (15 g), H_2O (7 ml) and a 0·1% aq. soln. of *m*-cresol purple (1 ml) and pack into a column for chromatography. Dissolve the sample ($\simeq 50$ mg) in H_2O (0·5 ml) and transfer it to the column with the aid of silicic acid (1 g) and $CHCl_3$ (10 ml). Develop with a 10% soln. of *n*-butanol in $CHCl_3$ (35 ml) at a rate of 2 or 3 ml per min., extrude the column, elute each acid separately from its section of the column with H_2O (30 ml), and titrate with aq. NaOH soln. to pH 8·5. Alternatively, develop in turn with a 10% soln. of *n*-butanol in $CHCl_3$ (65 ml), a 20% soln. of *n*-butanol in $CHCl_3$ (45 ml) and *n*-butanol (25 ml), and titrate each fraction with alcoholic NaOH, with *m*-cresol purple as indicator. Duplicate analyses may be completed by either method in 75 to 90 min. The accuracy and precision are ±1%. Interference due to copper is overcome by addition of a slight excess of oxalic acid to the sample. Interference due to HNO_3 is overcome by evaporation of the sample before analysis, but there is a simultaneous loss of 4 to 5% of the glutaric acid.

A. R. ROGERS

2283. Colorimetric determination of dicarboxylic acid derivatives as hydroxamic acids. V. Fishman-Goldenberg and P. E. Spoerri (Dept. Biochem., Hillside Hosp., Glen Oaks, N.Y., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1735–1737.—The study reported previously (*cf. Anal. Chem.*, 1958, **30**, 1327) of the reaction of derivatives of monocarboxylic acids with alkaline hydroxylamine is now extended to derivatives of dicarboxylic acids. The speed of reaction and the yield of colour depend on the number of carbon atoms between the carboxyl groups and whether the molecule is cyclic or acyclic.

A. R. ROGERS

2284. Titration of oxalic acid with cerium(IV) sulphate at room temperature using ferroin as internal indicator. V. Panduranga Rao and G. Gopala Rao (Andhra Univ., Waltair, S. India). *Talanta*, 1959, 2 (4), 370–375.—Contrary to previous statements (*cf.* Furman, *J. Amer. Chem. Soc.*, 1928, **50**, 755; Willard and Young, *Ibid.*, 1928, **50**, 1322), the reaction between $Ce(SO_4)_2$ and oxalic acid is quite rapid in the presence of HCl (0·2 to 6 N) even without the use of ICl as catalyst. Cerimetric titrations of oxalic acid (0·01 to 0·1 N) with ferroin as indicator can be made accurately at 20° to 25° if Ba^{2+} are present as a scavenger for SO_4^{2-}, which inhibit the reaction between oxalic acid and oxidised ferroin and between oxalic acid and Ce^{IV}. This modification of the Willard and Young method obviates titration at 50° (which causes some dissociation of ferroin) and also the use of ICl as catalyst. *Procedure*—To the sample soln. (5 to 10 ml) add conc. HCl (15 to 20 ml) plus 0·5 M aq. $BaCl_2$ (20 ml) and dilute to $\simeq 200$ ml (300 ml for

$\simeq 0·1$ N oxalic acid). Add 0·01 M ferroin (3 or 4 drops) and titrate with 0·05 M $Ce(SO_4)_2$, with dropwise addition towards the end, until the red colour is discharged.

W. J. BAKER

2285. Characterisation of glyoxylic acid in media arising from the action of periodic acid on organic compounds. P. Fleury and L. Le Dizet (Lab. de Chim. Biol., Fac. de Pharm., Paris, France). *Bull. Soc. Chim. France*, 1959, (7–8), 1186–1189.—Two modifications of existing methods of characterising glyoxylic acid (*cf.* Bougault, *Compt. Rend.*, 1909, **148**, 1270; Fosse and Hieulle, *Ibid.*, 1925, **181**, 286) are given. *Procedure*—(i) After the appropriate time of contact of the sample with excess of HIO_4, 1 drop in excess of ethanediol is added to reduce unchanged HIO_4 to iodate, which does not interfere. If necessary, formaldehyde must be eliminated by evaporation *in vacuo* with H_2SO_4 and then with NaOH - $KMnO_4$. The oxidised sample ($\equiv \simeq 6$ mg of glyoxylic acid in 4 ml of water) is treated with 2 ml of aq. NaOH soln., 0·2 ml of acetophenone and 2 ml of ethanol. The soln. is set aside for 6 hr. and extracted with diethyl ether (3 × 3 ml). The aq. layer is rejected, the ether layer is evaporated to dryness, and the residue is dissolved in 3 ml of water. After extraction with 2 ml of ether, the diphenacylacetic acid formed is pptd. from the aq. layer with acid, and identified by its m.p. and equiv. wt. (ii) Unchanged periodic acid in the sample is reduced to iodate with ethanediol as described above and the iodate is pptd. with $BaCl_2$ (acetate would interfere). The filtered soln. is treated with 100 mg of hydrazine sulphate and after 15 min. is centrifuged and 1 ml of acetic acid containing 5 mg of xanthhydrol is added. After 1 hr. in the dark the ppt. formed (even in the absence of glyoxylic acid) is centrifuged, drained and heated on a water bath for 15 min., becoming red if glyoxylic acid is present. The ppt. is suspended in ethanol and adjusted with *N* methanolic NaOH to pH 8. The absorption max. of the coloured soln. is at 520 to 540 mμ.

E. J. H. BIRCH

2286. Spectrophotometric determination of primary nitroparaffins by coupling with *p*-diazobenzenesulphonic acid. I. R. Cohen and A. P. Altshuller (Air Pollution Engng Res., Robert A. Taft Sanitary Engineering Center, U.S. Dept. of Health, Educ. and Welfare, Cincinnati, Ohio). *Anal. Chem.*, 1959, **31** (10), 1638–1640.—Mix a soln. of the sample (*e.g.*, <50 μg of nitromethane) in 10% aq. methanol (1 ml) with phosphate buffer of pH 4·3 (1·4 ml) and 3% KOH soln. (0·6 ml). Add a mixture of a 1·1% aq. soln. of KNO_2 (2·5 ml) and a 0·72% soln. of sulphanilic acid in 0·2 N HCl (2·5 ml) to the soln. with vigorous shaking, and measure the extinction at 440 mμ (for nitromethane) or at 395 mμ (for higher nitroparaffins) within 2 min. Perform a blank determination. The reaction is given by 2-ethyl-2-nitropropane-1 : 3-diol as well as by primary nitroparaffins, but not by secondary nitroparaffins or by 2-methyl-2-nitropropanol.

A. R. ROGERS

2287. Sub-micro methods for the analysis of organic compounds. VIII. Factors associated with sub-micro titration in glacial acetic acid. R. Belcher, J. Berger and T. S. West (Chem. Dept., The Univ., Birmingham). *J. Chem. Soc.*, 1959, (9–10), 2877–2882.—The use of $HClO_4$ in glacial acetic acid as titrant of organic bases on the sub-micro scale (vol. $\simeq 0·4$ ml; titrant required 0·03 to 0·05 ml) is

investigated. Potassium hydrogen phthalate is sufficiently soluble to be used as a primary standard. Evaporation of the solvent (largely round the plunger of the syringe) is troublesome and times for titrations should agree to within 3 to 5 min. Several indicators are investigated and crystal violet is preferred. It is necessary to titrate to a fixed colour by using a colour comparison soln. prepared by titrating urea in acetic acid with $HClO_4$ to a royal-blue colour with crystal violet. A blank titration ($\simeq 10\%$ of the titre) is always required and its value increases with vol. of solvent. The values of the potentiometric blank (with a glass electrode stored in glacial acetic acid vs. a silver - silver chloride electrode) and visual blanks are different, and the difference increases with vol. of solvent. The effect of water in the solvent is to decrease the sharpness of the end-point with very little effect on the amount of the blank. The water content should be > 0.15 to 0.20%. The thermal expansion of acetic acid is greater than that of water and a temp. correction is applicable. The coeff. of variation for potentiometric standardisations is $\pm 0.5\%$ and for visual standardisations is $\pm 0.7\%$, but the normalities differ by 1%. The stability of the reagents is discussed.

IX. Titration of organic bases and amine hydrohalides in glacial acetic acid. R. Belcher, J. Berger and T. S. West. *Ibid.*, 1959, (9-10), 2882-2885.—The sub-micro titration of bases in glacial acetic acid with $HClO_4$, described above, is applied to metal salts, S-benzylthiuronium salts and alkaloids, and results are compared with those obtained by the macro-method. For visual end-points, with crystal violet as indicator, the blue comparison soln. (*loc. cit.*), or for Li benzoate a blue - green comparison soln., is used. A precision of $\pm 1\%$ is attainable, but the macro-method is preferred. A back-titration method is used when the perchlorate is more soluble than the base. Amine hydrochlorides are titrated in the presence of Hg acetate, of which the amount in excess is not critical, but which reacts with the indicator and increases the blank. With aromatic amines some acetylation takes place, which reduces the titre unless the solvent contains 0.75% of water, and this adversely affects the end-point.   E. J. H. BIRCH

2288. Titration characteristics of organic bases in nitromethane. C. A. Streuli (Central Research Div., American Cyanamid Co., Stamford, Conn.). *Anal. Chem.*, 1959, **31** (10), 1652-1654.—Amines show normal behaviour on potentiometric titration with $HClO_4$ in nitromethane, but ureas, amides and mono-substituted amides show extremely steep titration curves in the region of 20% to 80% neutralisation. This behaviour is attributed to hydrogen bonding between solute molecules. Amides, ureas, heterocyclic bases and hydroxy-amines show relatively greater basicity in nitromethane than in water.   A. R. ROGERS

2289. N-(Hydroxymethyl)-3- and -4-nitrophthalimides as new reagents for the characterisation of amines. M. L. Vasa and K. C. Kshatriya (Chem. Dept., M. G. Science Inst., Ahmedabad). *J. Indian Chem. Soc.*, 1959, **36** (7), 527-528.—3-Nitro-(I) and 4-nitro-phthalimides (II) are condensed with primary aromatic amines in the presence of formaldehyde by the method of Winstead and Heine (*cf. Anal. Abstr.*, 1956, **3**, 1417) to yield the corresponding N-(arylaminomethyl)nitrophthalimides. The N-(hydroxymethyl) derivatives of I and II, prepared by the method of Winstead and Heine,

yield the same products with primary aromatic amines as do the parent compounds. Crystal colours, habits and m.p. are tabulated for 19 amine derivatives.   I. JONES

2290. Determination of small amounts of secondary amine in high-molecular-weight fatty primary amines. A. J. Milun and J. P. Nelson (Chem. Lab., General Mills Inc., Minneapolis 13, Minn., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1655-1657.—The primary amine is removed by reaction with salicylaldehyde (I) and the blue - green colour formed by interaction of the secondary amine with bromocresol green (II) is measured. The concn. of secondary amine (in the range $<1\%$) is calculated by use of a calibration curve. The reproducibility is estimated to be $\pm 0.05\%$ absolute. Tertiary amines in concn. $<1\%$ cause no interference. *Procedure*—Mix a soln. of the sample (50 mg) in *iso*propyl alcohol (2 ml) with a 5% soln. of I in *iso*propyl alcohol (2 ml), set aside for 10 to 30 min., add a 0.015% soln. of II in $0.0002\ N$ *iso*propyl alcoholic NaOH, dilute to 10 ml, and measure the extinction at 627 mμ.   A. R. ROGERS

2291. Analytical study of aminoethyl vinyl ether system based on countercurrent extraction. I. Rosenthal, F. Jackson and W. Watanabe (Rohm & Haas Co., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1702-1705.—Commercial samples of aminoethyl vinyl ether may also contain ethanolamine and the Schiff base of aminoethyl vinyl ether with acetaldehyde. The three components may be separated by 30-tube countercurrent distribution in the system dichloromethane - H_2O. The following method is simpler though less direct. *Procedure* —To determine total vinyl groups, dissolve the sample (10 milli-equiv.) in H_2O (10 ml) and titrate with $0.5\ N$ HCl; add $0.5\ N$ hydroxyammonium chloride (50 ml), heat at $85°$ for 2 hr. in a sealed vessel, cool, and titrate with $0.5\ N$ NaOH. To determine total base, dissolve the sample (0.3 g) in H_2O (50 ml) and titrate with $0.1\ N$ HCl. To determine ethanolamine, dissolve the sample (3 g) in a mixture of 10% (w/w) aq. NaCl (100 ml) and $0.1\ N$ NaOH (1 ml), and extract with dichloromethane (6×100 ml); wash each extract in succession with two further portions of 10% NaCl soln. (100 ml) - $0.1\ N$ NaOH (1 ml) and titrate the combined aq. layers with $0.1\ N$ HCl. Use bromocresol green as indicator in all titrations.
                                          A. R. ROGERS

2292. Photometric determination of quaternary ammonium compounds with hexanitrodiphenylamine. I. G. Schill and B. Danielsson (Royal Inst. of Pharm., Stockholm, Sweden). *Anal. Chim. Acta*, 1959, **21** (3), 248-254 (in English).—Pentanitrodiphenylamine (I), which may occur as an impurity in commercial hexanitrodiphenylamine (II), can be separated by partition chromatography, with a phosphate buffer (pH 7) on kieselguhr, with $CHCl_3$ as the mobile phase. The dissociation constants, partition constants and absorption spectra of the two pure compounds have been measured, and it has been demonstrated that, when used as a photometric reagent for quaternary ammonium salts, II must be free from I. The extraction of the complex between II and the quaternary ammonium salt by an organic solvent ($CHCl_3$ or dichloromethane) must be carried out at pH 10 to 11 to minimise the extraction of II, which has a significant extinction at the extinction maximum of the complex.   W. T. CARTER

2293. Bromatometric determination of some hydrazine derivatives. F. Jančik, O. Činková and J. Körbl (Res. Inst. Pharm. and Biochem., Prague). *Coll. Czech. Chem. Commun.*, 1959, **24** (8), 2695–2698 (in German).—The potentiometric titration of some hydrazine derivatives with bromate in an acid medium was studied and conditions were found for the determination of hydrazine (**I**), phenylhydrazine (**II**), semicarbazide (**III**), oxalic acid dihydrazide (**IV**), isoniazid (**V**) and cyanoacethydrazide (**VI**). *Procedure for* **I** *to* **V**—Dissolve the sample (4 to 60 mg of **I** sulphate, 5 to 73 mg of **II** hydrochloride, 4 to 57 mg of **III** hydrochloride, 4 to 62 mg of **V** or 2 to 31 mg of **IV**) in 3 to 4 N HCl (**I**, **IV** or **V**) or N HCl (**II** or **III**), add KBr (1 g) (not for **III**) and titrate potentiometrically with 0·1 N KBrO$_3$. *Procedure for* **VI**—Dissolve 4 to 60 mg of **VI** in N HCl (50 ml), add KBr (1 g) and titrate with 0·1 N KBrO$_3$, with shaking. When the first yellow colour appears, immediately add a 20% excess of 0·1 N KBrO$_3$ and KI (1 g), stopper the flask and set aside in the dark for 5 min. Then back-titrate with 0·1 N Na$_2$S$_2$O$_3$, with starch as indicator.

J. ZÝKA

2294. Quantitative determination of cyanohydrins. C. Berther, K. Kreis and O. Buchmann (Holzverzuckerungs A.-G., Domat/Ems, Switzerland). *Z. anal. Chem.*, 1959, **169** (3), 184–187 (in German).— The method is based on the reaction of CN⁻ with Ni²⁺ to form the [Ni(CN)$_4$]²⁻ complex. Cyanohydrins (1 to 2 millimoles) are treated with 0·1 M NiSO$_4$ (5 to 10 ml) and, after addition of H$_2$O, conc. aq. NH$_3$ (10 ml) and a little murexide, the excess of Ni²⁺ is back-titrated with 0·1 M EDTA. The colour change of the indicator is from yellow to reddish-violet. The method was used to follow the hydrolysis of cyanohydrins to α-hydroxy acids and the conversion of cyanohydrins into aminonitriles by means of ammonia.

G. P. COOK

2295. Quantitative determination of vinyl cyanide with piperidine. A. P. Terent'ev, M. M. Buzlanova and S. I. Obtemperanskaya (M. V. Lomonosov Moscow State Univ.). *Zhur. Anal. Khim.*, 1959, **14** (4), 506.—Add the sample of vinyl cyanide (30 to 50 mg) in a sealed ampoule to a 100-ml flask (with a ground glass stopper) containing 0·5 g of piperidine in 5 ml of dioxan. Break the ampoule with a glass rod, wash down the rod with dioxan, add 0·2 ml of 50% acetic acid, mix and set aside for 30 min. Add 5 ml of acetic anhydride dropwise from a burette to combine with excess of piperidine, then 3 drops of mixed indicator (0·2 g of methyl red and 0·04 g of methylene blue in 60 ml of methanol and 40 ml of water), and titrate the β-piperidinopropionitrile with 0·05 N HCl to an intense purple colour. Carry out a blank simultaneously. Acrylic acid and acrylates interfere; ethylene cyanohydrin does not.

C. D. KOPKIN

2296. Spectrophotometric determination of biuret in urea. J. Sverak (Österreich. Stickstoffwerke A.-G., Linz, Austria). *Z. anal. Chem.*, 1959, **169** (3), 178–184 (in German).—The method is based on the reaction of biuret with Ni tartrate in alkaline soln. to form a Ni - biuret complex, which gives absorption maxima at 465 and 260 mμ. The former absorption maximum is generally used, but for concn. of <0·5% of biuret the maximum at 260 mμ is more suitable. The coeff. of variation varies from ±2·58 to ±0·48% and the accuracy from +0·7 to −0·2% at the 1 and 5% biuret levels, respectively.

G. P. COOK

2297. New method for the determination of the esters of carbamic acid [urethanes]. Z. Tóth and I. Krasznai (United Pharm. and Foodstuff Factory, Budapest). *Magyar Kém. Foly.*, 1959, **65** (8), 289–291.—Urethanes lose NH$_3$ in the presence of Raney nickel containing 3 to 4% of Al. This reaction is quant. after boiling the compounds with 4 to 5% alkali for ≃30 min. Related compounds not containing the NH$_2$·CO·OR structure (R is alkyl), *i.e.*, substituted carbamic esters and derivatives of carbonic acid, do not react quantitatively. The procedure (described in detail) is similar to that for a Kjeldahl determination. The Raney nickel must be purified from N-containing impurities by boiling it with NaOH soln. (4 to 5%) for 20 min. In the cases studied (ethyl and butyl carbamates and meprobamate) the error was ±0·5%.

A. G. PETO

2298. Paper-chromatographic determination of phosphoric acid and its mono- and di-butyl esters. V. P. Shvedov and S. P. Rosyanov (Lensovet Technol. Inst., Leningrad). *Zhur. Anal. Khim.*, 1959, **14** (4), 507–508.—Irradiation of tributyl phosphate causes the formation of the di- and mono-butyl esters, which exert a considerable influence on the extraction of Th, U and their fission products. These esters and phosphoric acid may be separated by paper chromatography. Paper strips 2·5 cm wide were used, and 0·02 ml of a soln. of the acids was placed 2 cm from the edge and dried. The following mixtures of solvents were used— (i) butanol - methanol - water (4 : 1 : 2); (ii) isoamyl alcohol - pyridine - 25% aq. NH$_3$ (2 : 7 : 10); (iii) CHCl$_3$ - methanol - water (4 : 5 : 1); (iv) propanol - 25% aq. NH$_3$ - water (6 : 1 : 3); and (v) methanol - 25% aq. NH$_3$ - water (6 : 1 : 3). The chromatograms were sprayed with a mixture of 60% HClO$_4$ (5 ml), N HCl (10 ml) and 4% ammonium molybdate soln. (25 ml) made up to 100 ml with water, dried in warm air, heated at 85° for 7 min., set aside in the air to become saturated with moisture, and finally suspended in a vessel containing H$_2$S for 5 or 10 min. The developed spots were cut out, ashed with a mixture (1 : 1) of conc. H$_2$SO$_4$ and HNO$_3$ and the orthophosphate was determined. If CHCl$_3$ satd. with water is used as chromatographic solvent, the dibutyl ester is not displaced from its point of introduction; this may be used to separate it from tributyl phosphate.

C. D. KOPKIN

2299. New colour reaction for thiosemicarbazide with sodium nitroprusside. V. G. Belikov (Pyatigorsk Pharm. Inst.). *Zavod. Lab.*, 1959, **25** (8), 931.—For a macro-test, 1 ml of the thiosemicarbazide soln. in 10% aq. NH$_3$ is mixed with 2 ml of 1% sodium nitroprusside soln. and the red soln. obtained is treated with 0·5 ml of glacial acetic acid. If ≮100 μg of thiosemicarbazide is present the soln. turns yellowish green and then, after 2 to 3 min., reddish violet. The limiting dilution is 1 in 1 × 10⁴. In the absence of thiosemicarbazide the soln. is finally colourless. For a spot test on porcelain, one drop of the soln., one or two drops of the reagent and one drop of glacial acetic acid are used. The minimum amount detectable is 4 μg, and the limiting dilution is as before.

G. S. SMITH

2300. Tetracyanoethylene as a colour-developing reagent for aromatic hydrocarbons. P. V. Peurifoy, S. C. Slaymaker and M. Nager (Houston Res. Lab., Shell Oil Co., P.O. Box 2527, Houston 1, Tex., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1740.—To

detect microgram quantities of aromatic hydro-carbons on paper chromatograms, spray with a saturated soln. of tetracyanoethylene in benzene. Of a wide variety of aliphatic and aromatic hydro-carbons tested, only the alkylbenzenes and poly-nuclear aromatics give coloured spots. Most of the colours fade on heating.　　　　A. R. ROGERS

2301. **Isatin tests for aromatic hydrocarbons and phenols.** E. Sawicki, T. W. Stanley, T. R. Hauser and R. Barry (Air Pollution Engng Res., Robert A. Taft Sanitary Engng Center, U.S. Dept. of Health, Educ. and Welfare, Cincinnati, Ohio). *Anal. Chem.*, 1959, **31** (10), 1664–1667.—The reaction of certain isatin derivatives with PCl_5 yields isatin chlorides which readily give cationic indigoid dyes with phenols and polynuclear aromatic hydrocarbons. The reagents are prepared by heating the isatin derivatives under reflux with PCl_5 in either benzene or *o*-dichlorobenzene. To a soln. of the hydro-carbon ($\simeq 5 \mu g$) in $CHCl_3$ (1 ml) add trifluoroacetic acid (2 ml) and $POCl_3$ (0·5 ml), shake gently and set aside for 5 min.; add the reagent soln. (1 ml), dilute with $CHCl_3$ to 10 ml and after 5 min. measure the absorption spectrum. The long-wavelength maxima and sensitivities are tabulated for the products of interaction of pyrene, benzo[*a*]pyrene, benzo[*e*]-pyrene, perylene, benzo[*ghi*]perylene and anth-anthrene with the reagents prepared from 10 isatin derivatives. The tests have been applied to the semi-quant. determination of *peri*-condensed aro-matic hydrocarbons in the particulate matter of air (*cf.* Sawicki *et al.*, *Anal. Abstr.*, 1958, **5**, 4197).
A. R. ROGERS

2302. **Determination of C9 to C10 alkylbenzenes.** S. H. Hastings and D. E. Nicholson (Humble Oil and Refining Co., Baytown, Tex.). *Anal. Chem.*, 1960, **32** (2), 299.—Infra-red data are given for indane, *o*-cymene, 1:3-diethylbenzene and *n*-butyl-benzene.　　　　N. E.

2303. **Determination of ethylbenzene in ethyl-nitrobenzene.** O. S. Vladýrchik, L. L. Bespalova and P. M. Kochergin. USSR Pat. No. 118,652; 10 March, 1959.—Ethylbenzene (**I**) in ethylnitro-benzene (**II**) is determined by the amount of HNO_3 used in nitrating it to **II** with a mixture of HNO_3 and H_2SO_4, after forming the coloured complex of **I** with nitrosylsulphuric acid. *Procedure*—Weigh 0·5 to 0·6 g of technical neutral **II** into a conical flask, add 20 ml of nitrosylsulphuric acid (*e.g.*, containing 85·21% of H_2SO_4 and the equiv. of 0·26% of N_2O_3); an intense red, or, with low concn. of **I**, a pink colour is formed. Rapidly titrate the soln. with a nitrating mixture (*e.g.*, 81·50% of H_2SO_4 and 0·4% of HNO_3) till colourless. The percentage content of **I** is then 100 VT/a, where V is the vol. of nitrating mixture used, T is the factor for conversion to **I** and a is the wt. of sample; T is found by titration of the HNO_3 in the mixture with 0·1 N $FeSO_4$, and multiplication of the normality by 106·16 (mol. wt. of **I**).
C. D. KOPKIN

2304. **Tetrahydrofuran - water mixture as a polaro-graphic solvent. Determination of the lower poly-phenyls.** L. Silverman, W. G. Bradshaw and M. E. Shideler (Atomics International, North American Aviation Inc., Canoga Park, Calif.). *Anal. Chem.*, 1959, **31** (10), 1669–1671.—Tetrahydrofuran may be purified by passage through a column of activated alumina - Celite (3:1) and may be stored for

<24 hr. over activated alumina. The mixture tetrahydrofuran - H_2O (3:1) has a good solvent action for supporting electrolytes such as tetra-butylammonium iodide and for many organic compounds, and may be used at potentials as negative as $-2·5$ V *vs.* the mercury-pool anode. The diffusion currents obtained for organic com-pounds in this mixture are larger than those in aq. dioxan. Biphenyl and the terphenyls can be quant. determined, and reduction waves have also been obtained for 4-bromobiphenyl, nitrobenzene, naphthalene, triphenylene, 2-bromonaphthalene, anthracene and pyrene.　　　　A. R. ROGERS

2305. **Paper chromatography of phenols with polyamide-impregnated paper.** Kung-Tsung Wang (Dept. of Chem., Nat. Taiwan Univ., China). *J. Chinese Chem. Soc., Ser. II*, 1959, **6** (1), 73–79 (in English).—Paper treated with a 5% soln. of polycaprolactam in formic acid (Carelli *et al.*, *Nature*, 1955, **176**, 70) was used to separate naturally occurring phenols. The R_F values of 14 phenols in 7 different solvents are tabulated.

G. S. ROBERTS

2306. **Ultra-violet absorption spectra of phenols.** N. I. Shergina, V. P. Kuznetsova, A. S. Nachmano-vich and I. V. Kalechits. *Optics and Spectroscopy*, 1959, **6** (6), 524–526; English translation of *Optika i Spektroskopiya*, 1959, **6**, 803.—Data are given for the absorption spectra of 31 phenols. The influence of substituents on the position and intensity of the absorption bands has been studied.　　　　N. E.

2307. **Observations concerning the adsorption affinities of dihydroxybenzenes and pyrogallol on strong anion exchangers in hydrogen sulphite form.** T. Enkvist and J. Halmekoski (Dept. Chemistry, Univ. of Helsinki, Finland). *Suomen Kem., B*, 1959, **32** (9), 173–175 (in English).—A strongly basic resin in HSO_3^- form was used for separation of some lignin-cleavage phenols, which are formed by heating lignin or spent liquors with alkaline reagents. The experiments carried out with 50% methanol as eluent showed that the resin adsorbs pyrogallol, catechol, quinol, and, to a less extent, resorcinol, whereas guaiacol and 4-acetylguaiacol readily pass through the column.　　　　E. SJÖSTRÖM

2308. **Determination of polymethylbenzyl ben-zoates.** D. E. Nicholson (Humble Oil & Refining Co., Baytown, Tex.). *Anal. Chem.*, 1960, **32** (2), 298.—Infra-red data are given for 2:4:5-trimethyl-benzyl 2:4:5-trimethylbenzoate, 2:4-dimethyl-benzyl 2:5-dimethylbenzoate and 2:4-dimethyl-benzyl 2:4-dimethylbenzoate.　　　　N. E.

2309. **The polarography of lead styphnate.** L. R. Leake and G. F. Reynolds (Chemical Inspectorate, Royal Arsenal, Woolwich, London). *Anal. Chim. Acta*, 1959, **21** (3), 255–260 (in English).—By a combination of normal polarography and derivative polarography with a.c. superimposition it is possible to determine lead and styphnic acid (2:4:6-trinitroresorcinol) in the same soln. The polaro-grams are measured at pH 1·8 (Na citrate - HCl buffer) against a mercury-pool anode, with proteose peptone as maximum suppressor. The styphnic acid is measured on the normal polarogram from the wave at $-0·1$ to $-0·3$ V, and the lead on the derivative polarogram from the peak at $-0·5$ V. The error for either component does not exceed $\pm 5\%$.　　　　W. T. CARTER

2310. Rapid determination of p-dimethylamino-acetophenone by direct titration. Hsin-Yün Yü and Teng Tao. *Chem. World*, 1959, **14** (7), 342–343.—*p*-Dimethylaminoacetophenone may be directly titrated by using acetic anhydride instead of acetic acid as the solvent. The titration may be carried out potentiometrically, with $0.2 N$ $HClO_4$, or with crystal violet as indicator. The end-point is determined by measuring the values of $\Delta E/\Delta V$. The best solvent is a mixture of glacial acetic acid and acetic anhydride (1:1). The procedure takes only 20 to 30 min. and is accurate and economical.　　SCI. ABSTR. CHINA

2311. Organic analysis. XIV. Infra-red spectra of phenylsulphonyl derivatives. (3). The C–H deformation vibrations of the benzene ring, the CH_3 rocking frequencies of the SO_2CH_3 group and the characteristic absorption bands of the SO_2NH_2 group. Tsutomu Momose, Yo Ueda and Tatsuo Shoji (Pharm. Inst., Med. Fac., Univ. Kyushu). *Chem. Pharm. Bull., Tokyo*, 1959, **7** (6), 734–739 (in English).—The C–H in-plane deformation vibrations of the benzene ring may be intensified by the polar and heavy SO_2 group in phenylsulphonyl compounds. The CH_3 rocking vibrations in the SO_2CH_3 group have smaller wave numbers than that of dimethyl sulphide and are in the region of 980 to 950 cm^{-1} and 790 to 760 cm^{-1}; RSO_2NH_2 compounds have characteristic frequencies in the region 919 to 896 cm^{-1}, which may be assigned to the S–N stretching vibration.

2312. Determination of C_{10} to C_{11} naphthalenes. D. E. Nicholson and S. H. Hastings (Humble Oil & Refining Co., Baytown, Tex.). *Anal. Chem.*, 1960, **32** (2), 298.—Infra-red data are given for naphthalene, 1-methylnaphthalene, 2-methylnaphthalene and 1:2:3:4-tetrahydronaphthalene.　　N. E.

2313. Thermochromic detection of polynuclear compounds containing the fluorenic methylene group. E. Sawicki and W. Elbert (Taft San. Engng Center, U.S. Dept. of Health, Education and Welfare, Cincinnati, Ohio). *Chemist Analyst*, 1959, **48** (3), 68–69.—The fluorene compound is dissolved in dimethylformamide and the soln. is made alkaline with tetraethylammonium hydroxide soln. The resulting compound is oxidised to the fluorenone by shaking in air and is then reduced by boiling with potassium borohydride, forming the highly coloured fluorenol anion. The colour fades on cooling and shaking. Inner-ring polycyclic *p*-quinones and fluorenones give a positive reaction, but can be distinguished by their thermochromic behaviour in the absence of tetraethylammonium hydroxide (*cf.* Sawicki *et al.*, *Anal. Chem.*, 1958, **30**, 2005).
　　　　　　　　　　　　　　G. S. ROBERTS

2314. Quantitative determination of α-pinene. R. L. Kenney, T. C. Singleton and G. S. Fisher (Naval Stores Res. Sta., Southern Utilization Res. and Development Div., Agric. Res. Service, U.S. Dept. of Agric., Olustee, Fla.). *Anal. Chem.*, 1959, **31** (10), 1676–1678.—This new method is closely analogous to isotope dilution methods; it depends on optical activity rather than on radioactivity and has been named "optical dilution." The ratio of (+)- to (−)-α-pinene in a sample of turpentine can be calculated from the optical rotation of a small amount of pure α-pinene isolated from the mixture; quant. separation of all of the α-pinene is not necessary. Similarly, the ratio of the (unknown) concn. of α-pinene of any rotation originally present

in a sample of turpentine to the (known) concn. of an added α-pinene of different rotation can be calculated from the rotation of the pure α-pinene distilled from the mixture. The concn. of α-pinene in known mixtures with β-pinene was determined with an accuracy of $\pm0.5\%$ (P = 0.9).
　　　　　　　　　　　　　　A. R. ROGERS

2315. Determination of anthraquinonesulphonic acids. V. V. Kozlov and A. A. Davýdov (G. V. Plekhanov Inst. of National Economy). *Zavod. Lab.*, 1959, **25** (8), 926–928.—The gravimetric determination of anthraquinonesulphonic acids as chloroanthraquinones suffers from the prolonged time requisite for the chlorination by $KClO_3$. Conditions for considerably reducing this time are described. Thus if a mixture of 1.3 g of the potassium salt of anthraquinonesulphonic acid, 80 ml of 2 N HCl and 70 ml of water is boiled in a three-necked flask with stirrer and reflux condenser, and treated with a boiling soln. of 0.9 to 1.0 g of $KClO_3$ in 50 ml of water, the reaction is completed when the contents of the flask have been boiled for 1.5 hr. The ppt. is filtered off after the reaction mixture has been cooled to between 60° and 70°, then washed with hot water, and dried at 100°. Conditions are also given for the chlorination by $K_2Cr_2O_7$ in the presence of HCl.　　　　　　G. S. SMITH

2316. Paper electrophoresis of nicotinic acid derivatives. T. K. Sundaram, K. V. Rajagopalan and P. S. Sarma (Univ. Biochem. Lab., Madras, India). *J. Chromatography*, 1959, **2** (5), 531–535 (in English).—Nicotinic acid, nicotinamide, nicotinuric acid, N^1-methylnicotinamide, diphosphopyridine nucleotide and triphosphopyridine nucleotide are separated in 1 or 2 hr. by electrophoresis on a 30-cm strip of Whatman No. 1 filter-paper at a potential difference of 280 V in 0.033 M borate buffer of pH 8.9 or 0.05 M barbiturate buffer of pH 8.6.　　　　　　A. R. ROGERS

2317. Mass spectra of alkylindoles. J. H. Beynon and A. E. Williams (Dyestuffs Div., I.C.I. Ltd., Manchester, England). *Appl. Spectroscopy*, 1959, **13** (4), 101–105.—Data are given for 11 alkylindoles. Correlations between their structures and mass spectra are given, and peaks characteristic of alkylindoles are discussed.　　K. A. PROCTOR

2318. Gas-chromatographic analysis of gases containing hydrocarbons. G. Scharfe (Farbenfabriken Bayer A.-G., Leverkusen). *Erdöl u. Kohle*, 1959, **12** (9), 723–728.—Apparatus has been developed for the routine chromatographic analysis of cracking gases. Twelve analysing units can each be connected to one of four recorders. The columns contain different stationary phases and each is used for the separation of a different group of gases. Data are tabulated for the analysis of a 40-component mixture.　　　　　G. BURGER

2319. Spectrophotometry in the near infra-red and its application to the analysis of hydrocarbon mixtures. G. Geppert and L. Kipke (Inst. org. Grundstoffchem., Leipzig). *Chem. Tech., Berlin*, 1959, **11** (8), 427–430.—Benzole from a pilot-scale fluidised-bed carbonising plant was redistilled into three fractions with cuts at 95°, 122° and 150°, and the height of the absorption peak of each fraction at the second aromatic overtone CH band at $\simeq 1.15$ mμ was determined by a twin-beam spectrophotometer, with a 1-cm cell, a glass or quartz prism, an ordinary filament lamp as source, and a

lead sulphide photocell for recording. The aromatic contents of the fractions were read from graphs of concn. vs. extinction for synthetic mixtures of benzene with hexane, toluene with octane, and xylenes with nonane; these are rectilinear and obey Beer's law. Differences in absorption by the xylenes are unimportant, but the calibration curve does not apply if ethylbenzene is present in the xylene fraction. The method is quicker than the ndM method and the relative error is estimated to be $< \pm 5\%$.   A. R. PEARSON

2320. Determination of the petrol content of natural gas by means of chromatographic separation and micro-combustion. J. Tóth and L. Gráf (Sci. Lab. for the Winning of Petroleum, Nagykanizsa, Hungary). *Magyar Kém. Foly.*, 1959, **65** (8), 324–328.—Petrol is defined as a mixture of hydrocarbons not lower than pentane. A detailed description is given of the apparatus, which consists of a gas-chromatographic column (usually 140 mm long with a diameter of 7 mm and packed with silica gel) and a combustion system, followed by Pregl-type absorption tubes. The C_2 or higher hydrocarbons can be burnt and thus determined qual. or quant., separately or in groups. In the determination of petrol, the hydrocarbons containing < 5 C atoms are collected and burnt together. The carrier gas is air at a velocity of 0·7 ml per sec. By this method, the petrol content of a 100-ml sample, containing 5 to 200 g of petrol (at n.t.p.) per cu. metre, can be determined with an error of $\pm 1·5\%$.   A. G. PETO

2321. Analysis of light hydrocarbons in crude petroleum by means of vapour-phase chromatography. J. van Rysselberge (Centre de Rech., Groupe Pétrofina, Brussels 12, Belgium). *Ind. Chim. Belge*, 1959, **24** (9), 1023–1036.—The gas chromatography of C_2 to C_5 hydrocarbons is effected with a crude petroleum by the use of a "pre-column" that removes the higher hydrocarbons. *Procedure* —The oil for sampling is kept under a serum cap in a bottle kept full by means of a mercury reservoir. It is withdrawn by means of a syringe, weighed and made up to 5 ml with diethyl ether. The columns used are a pre-column (6·35 mm × 13 to 40 cm) loaded with Celite 545 (retained by B.S. 120 mesh) impregnated with Apiezon grease, and provided with a by-pass, and a main column (8 mm × 2 metres) filled with Stercharmol (25 to 85 mesh) impregnated with 25% of dinonyl phthalate, so arranged that together with the katharometer it can be placed in a thermostat. The by-pass is provided with an adjusting valve so that the gas flow is the same through the pre-column and the by-pass. The prepared sample (0·3 to 0·4 ml) is injected at the top of the pre-column and hydrogen gas is passed for 4 to 5 min. The pre-column is then by-passed by means of magnetic valves and the passage through the main column is continued for 30 min., which enables hydrocarbons up to C_5 to register on the katharometer (bridge circuit described). Examples of the method are given. The conditions are— carrier gas (hydrogen) pressure 0·32 kg per sq. cm, gas flow 30 cu. cm per min., temp. 23·5°.   E. J. H. BIRCH

2322. Analysis of petroleum products in the C_{12} to C_{20} range. Application of FIA [fluorescent indicator adsorption] separatory and low-voltage massspectrometric techniques. G. L. Kearns, N. C. Maranowski and G. F. Crable (Gulf Res. and Development Co., Pittsburgh, Pa., U.S.A.). *Anal.*

Chem., 1959, **31** (10), 1646–1651.—A combination of fluorescent indicator adsorption techniques with standard and low-voltage mass spectrometry is used to analyse petroleum products in the C_{12} to C_{20} range. Examples are given of the application of the complete procedure to the determination of compositions of jet fuel, kerosine, Diesel oil and fuel oil. Low-voltage mass-spectra of aromatics indicate a relationship between sensitivity and the number of substitutions on the benzene ring.   K. A. PROCTOR

2323. Determination of sulphur compounds in ARAMCO crude oil. Spectrophotometric determination of aliphatic sulphides in the naphtha fraction. Yuzo Koga, Masao Kajiwara and Masaru Kawaguchi (Tia Nenryo Kogyo Co., Sodeshi, Iohara-gun, Shizuoka-ken). *J. Japan Petrol. Inst.* 1959, **2** (5), 488–491.—The extinction coeff. of the iodine adducts of aliphatic sulphur compounds at 308 mμ (Hastings, *Anal. Chem.*, 1953, **25**, 420; Karchmer, *Ibid.*, 1958, **30**, 80) obey Beer's law for < 20 p.p.m. of S per 50 ml for each compound, and the value remains unchanged for 1 hr. Since the position of the absorption spectrum differs slightly according to the compound, measurement of the area of the absorption curve between 280 and 370 mμ is more suitable for the determination of S in petrol fractions. The sample is diluted with *iso*octane and mixed with iodine in *iso*octane (0·1% w/v).   K. SAITO
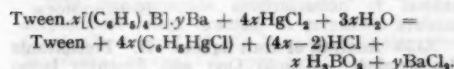
2324. Determination of a minute amount of water in petroleum products by the Karl Fischer method. Atsuhiko Tsuchiya and Yasuko Osawa (Res. Lab., Mitsubishi Oil Co., Oogimachi, Kawasaki, Kanagawa-ken). *J. Japan Petrol. Inst.*, 1959, **2** (1), 21–26.—The Karl Fischer method was modified by the use of a mixture of pyridine and ethanediol (5:1) so that the limit of determination could be extended to $\simeq 10$ p.p.m. Since petroleum products sometimes contain a small amount of aldehyde or ketone, the amount of methanol in the reagent is decreased. The results are lower than those obtained by the usual Karl Fischer method, but appear to be correct, because the samples contain carbonyl compounds which condense with methanol to give water.   K. SAITO

2325. Gravimetric method for analysing blastfurnace top gas. D. J. Kusler (U. S. Bur. Mines, Pittsburgh, Pa.). *U.S. Bur. Mines, Rep. Invest.*, 1959, No. 5452, 12 pp.—A primary or direct, continuously sampling, and automatically averaging gravimetric method for analysing gaseous mixtures of CO_2, CO, H and (by vol. measurement) N is described. The gaseous mixture is passed at a controlled and definite rate through a system that permits the CO_2 and H_2O retained by appropriate absorbents to be determined and the vol. of the residual gas (essentially N) to be measured. The accuracy and dependability have been established by comparison with very carefully conducted Orsat analyses. Methane and O are not determined.   CHEM. ABSTR.

2326. Infra-red spectra of asphalts [bitumens]. B. D. Beitchman (Nat. Bur. of Standards, Washington, D.C., U.S.A.). *J. Res. Nat. Bur. Stand., A*, 1959, **63** (2), 189–193.—The preparation of thin films (0·5 to 2·5 mil) of roofing asphalt (air-blown bitumen) is described, and their i.r. transmittances (τ) between 2·5 and 15 μ are reported and discussed

for 28 samples. Durability can be correlated with changes in carbonyl and hydroxyl absorption and also, for bitumen exposed initially to u.v. radiation, with changes in log τ^{-1} at the wavelengths of 5·88, 8·66, 9·71 and 2·91 μ.   W. J. BAKER

2327. Quantitative determination of the fatty acid monoesters of $\alpha : \omega$-dihydroxypolyoxyethylene sorbitan [Tweens] with sodium tetraphenylboron. R. Neu (Firma Dr. Willmar Schwabe G.m.b.H., Karlsruhe, Germany). *Arzneimittel-Forsch.*, 1959, **9** (9), 585–587.—With Na tetraphenylboron (**I**) and $BaCl_2$ Tweens form insol. complexes of the general formula Tween.$x[(C_6H_5)_4B].y$Ba. On treatment with aq. $HgCl_2$ soln. these decompose according to the scheme—

Tween.$x[(C_6H_5)_4B].y$Ba + $4x$HgCl$_2$ + $3x$H$_2$O =
Tween + $4x(C_6H_5HgCl)$ + $(4x-2)$HCl +
x H$_3$BO$_3$ + yBaCl$_2$.

Determination of Tween—Dissolve the sample (0·1 g) in H_2O (10 ml), add 10% aq. $BaCl_2$ soln. (1 ml) and heat on a boiling-water bath for 15 min. Cool, add 3% aq. **I** soln. (10 ml), mix and set aside for 14 hr. Filter off the ppt. in a sintered glass crucible with suction, wash it with H_2O (100 ml) and dry it to constant wt. *in vacuo* at 50°. *Analysis of the complex*—Dissolve the dried complex (0·1 g) in acetone (10 ml) and add, in the order given, methanol (40 ml), $HgCl_2$ (3 g), NaCl (1 g) and H_2O (120 ml), stopper the flask and stir magnetically for 1 hr. at room temp. Then add 0·5% ethanolic bromocresol purple soln. (2 drops) and titrate with 0·1 N NaOH to the end-point. This measures the HCl. Then add sorbitol (10 g) and titrate again with 0·1 N NaOH. This measures the H_3BO_3. Then acidify the soln. with dil. HCl, filter off the pptd. C_6H_5HgCl (**II**), wash it with dil. HCl, and remove the methanol from the united filtrate and washings on a water bath. Filter off the further ppt. of **II**, add to the filtrate and washings 2 N H_2SO_4 (10 ml) and digest on a water bath. Filter off the pptd. $BaSO_4$, wash it with aq. acetone and then with H_2O, and ignite and weigh it in the usual manner. From the results the composition of the complex and the factor can be calculated, and the Tween identified. Results are quoted for Tweens 20, 40, 60 and 80, in which $x:y = \simeq 2:1$.

A. G. COOPER

2328. Colorimetric determination of sodium lauryl sulphate. H. D. Graham and R. McL. Whitney (Dept. of Food Technol., Univ. of Illinois, Urbana, U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1673–1676.—Sodium hypochlorite and o-tolidine react at pH 3·5 to form a haloquinone which couples with sodium lauryl sulphate (**I**) at pH 3·0 to give a purple colour, the intensity of which is proportional to the concn. of **I**. *Procedure*—Shake thoroughly 3 N phosphate buffer of pH 3·5 (27 ml) with an aq. soln. of sodium hypochlorite containing 300 p.p.m. of available chlorine (1 ml) and a 0·1% soln. of o-tolidine dihydrochloride (2 ml). Two minutes after the addition of the o-tolidine add an aq. soln. of the sample (50 ml containing <30 p.p.m. of **I**), shake, set aside for 10 min. and measure the extinction at 590 mμ, with the buffer soln. as blank. To determine **I** bound by protein material in bacterial cells, hydrolyse the sample by boiling with 0·01 M Na_2HPO_4, filter through alkali-treated asbestos, and treat the acidified filtrate as described above.

A. R. ROGERS

2329. Use of gas chromatography in the analysis of volatile oils. D. Lamparsky (Res. Dept., L. Givaudan & Cie, S.A., Vernier-Genf). *Riechstoffe u. Aromen*, 1959, **9** (7), 201–206; 241–245.—For the determination of a constituent a working temp. of 20° to 50° below the b.p. of the constituent is used. For a complete analysis the oil may first be fractionated by normal methods and the fractions analysed by gas chromatography. For simple commercial determinations the internal standard method can be used: the sample is mixed with a known quantity of a suitable substance before subjection to gas chromatography, and the amount of the constituent to be determined is assessed from the ratio of the area of the elution peak of the constituent to that of the standard. A simple method of calculation is given. The standard must not be a constituent of the oil, and its elution peak must not interfere with the peaks of the oil constituents. Acetophenone is a very useful standard for use in determining citral a (geranial) and citral b (neral) in lemongrass oil; each citral gives its own peak so that its content can be determined from a single chromatogram. The results of the internal standard method are accurate to within 0·5% and are satisfactory for ordinary commercial purposes. The method is applied to the analysis of citronella oils for content of citronellal, citronellol and geraniol, with benzyl acetate as internal standard; clove oils for eugenol; and thyme oils for thymol and carvacrol (and other components).

H. L. WHITEHEAD

2330. Vanadametry. Vanadametric determination of phthalocyanines. G. Gopala Rao and T P. Sastri (Dept. of Chem., Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1959, **169** (1), 11–16 (in English).—Optimum conditions for the determination of metal-free phthalocyanine (**I**), Cu phthalocyanine (**II**) and Cu phthalocyaninetetrasulphonic acid (**III**) with $NaVO_3$ have been determined. **I** and **II** are resistant to oxidation, but can be obtained in a reactive form by trituration with syrupy H_3PO_4. This stage can be omitted with **III**. The dispersed pigment (60 mg) is then treated with 20 ml of 0·05 N $NaVO_3$ and enough H_2SO_4 to make the soln. 10 N, 6 N and 2 N for **I**, **II** and **III**, respectively. The vol. is made up to 60 ml and the mixture heated on a boiling-water bath for 60, 30 and 5 to 10 min. for **I**, **II** and **III**, respectively. After cooling, the excess of vanadate is determined by titration with standard $(NH_4)_2SO_4.FeSO_4.6H_2O$ soln. Typical results are tabulated.

S. M. MARSH

2331. Process and device for testing solids, liquids or gases for the presence of substances forming azo dyes. VEB Leuna-Werke "Walter Ulbricht" [Inventors: H. Bernzott and A. Meckelburg]. Brit. Pat. 818,845; date appl. 6.12.55.—The presence of phenols or of aromatic amines (*e.g.*, in waste effluents or in the atmosphere of workshops) is detected by the continuous production, at 0° to 40°, of diazo compounds from the amines and their coupling with the phenols to form azo dyes, which are detected visually or with the aid of a photoelectric cell combined with an acoustic or optical signalling device.   J. M. JACOBS

2332. Determination of carbonyl groups in modified celluloses. A comparison of the borohydride and copper number methods. S. Lidman-Safwat and O. Theander (Svenska Träforskningsinst., Stockholm). *Svensk Papp-Tidn.*, 1958, **61** (2),

42–46.—The borohydride method (Lindberg *et al.*, *Ibid.*, 1952, **55**, 13; 1954, **57**, 83) for the determination of carbonyl groups in sugars and oxidised cellulose was applied to dry-milled, acid-, hypochlorite- and chromate-treated cellulose and to some low-mol.-wt. carbohydrates (D-glucose, cellobiose, methyl β-D-6-, methyl β-D-3- and methyl β-D-2-oxoglucopyranosides, 5-oxo-, 2-oxo- and D-gluconic acids, methyl β-D-glucopyranoside and cellobi-itol. Comparison with results obtained by the copper number method (Centr. Lab. Swedish Cellulose Ind. Standards CCA3, 1940) shows that in the selected samples there is a linear relationship between the copper number and the carbonyl content as determined by the borohydride method. The numerical value of the ratio of the copper number to the carbonyl content varies with different types of cellulose. Results on low-mol.-wt. carbohydrates indicate that the reduction of celluloses having different structural composition will give different ratios. The stabilities of celluloses towards reduction with borohydride and copper reagents vary. The non-stoicheiometric copper numbers can be interpreted in terms of corresponding carbonyl contents and the method is suitable for the comparison of the reducing power of cellulose samples, modified under similar conditions.

B. RÖNNHOLM

2333. Determination of reducing sugars in sulphite waste liquors. K. Sato, Y. Matsumura, K. Miyakawa and S. Yoshimura (Women's Med. Coll., Shijuku-ku, Tokyo). *Svensk Papp.-Tidn.*, 1958, **61** (3), 68–71 (in English).—Ligninsulphonic acid can be pptd. with cationic surfactants [*e.g.*, cetyltrimethylammonium bromide (**I**) or benzyloxyethyl-lauryldimethylammonium bromide]. Complete pptn. occurs in a narrow surfactant concn. range which is dependent on the pH and neutral salt concn. *Procedure*—Dilute the sample (100 ml) with water (100 ml), add 6 *M* HCl (4 ml) and heat on a water bath under reduced pressure for 10 min. Cool, filter, wash the ppt. with water and dilute the filtrate to 250 ml. Take 1 ml of this soln. and dilute to 50 ml for the test soln. Add amounts (0·1 to 0·5 ml) of a 1% aq. soln. of **I** to five testtubes each containing 1 ml of the test soln. Shake, set aside for 10 min., and note in which tube the pptn. appears to be complete. Suppose this was the case in the tube containing 0·2 ml of **I** soln., then add 1% aq. soln. of **I** (1·5, 2·0 and 2·5 ml) to three portions (10 ml) of the test soln., shake, set aside for 10 min. and select the tube in which the pptn. is apparently most complete. Separate the ppt. and use the soln. for colorimetric sugar determination by the methods of Somogyi (*J. Biol. Chem.*, 1945, **160**, 61) or Roe (*Ibid.*, 1955, **212**, 335). The surfactants used do not interfere. No significant adsorption of glucose on the ligninsulphonic acid ppt. was observed. The recoveries of glucose added to waste liquor were 98 to 104%. Results for the determination of mannose, galactose, xylose and fructose by different colorimetric procedures are presented. B. RÖNNHOLM

2334. Determination of calcium in sulphite spent liquor. O. Franzon, G. Ivarsson and O. Samuelson (Chalmers Tekn. Högskola, Göteborg, Sweden). *Svensk Papp-Tidn.*, 1958, **61** (6), 165–166 (in English).—Calcium is separated on a cation-exchange resin and eluted with EDTA soln. (**I**), the excess of which is titrated with standard MgSO₄ soln. *Column*—The resin (Dowex 50) in its NH₄⁺ form (particle size 0·3 to 0·4 mm) is used in a

column of diam. 10 mm and height ≃200 mm, at a flow-rate of ≃2 ml per sq. cm per min. *Reagents* —(*i*) Standard soln. of **I** (0·1 *M*) containing 10% (v/v) of buffer soln. (*ii*) Buffer soln. (pH 10) containing 70 g of NH₄Cl and 570 ml of aq. NH₃ (sp. gr. 0·9) per litre. *Procedure*—Pass the test soln. (<4 millimoles of Ca) through the column and wash with de-ionised water. Elute with dil. buffer soln. (1:20, 25 ml) and then with sufficient **I** to give a mol. ratio of **I** to Ca of >1·25. Wash the column with dil. buffer soln. (1:20) (4 × 25 ml). Dilute the eluate and washings to 200 ml, add buffer soln. (10 ml) and 4 to 6 drops each of Eriochrome black T and methyl red soln. (1% in ethanol). Titrate with standard MgSO₄ soln. to a grey colour. Recoveries of ≮99·5% of added Ca and reproducibilities better than ±0·5% (relative) are reported.

B. RÖNNHOLM

2335. Analysis of kraft liquors. II. Precipitants for sulphide. Kazuki Ono and Zenshiro Isono (Fac. of Agric., Univ. of Osaka Prefecture, Sakai, Osaka). *J. Jap. Tech. Ass. Pulp Paper Ind.*, 1959, **13** (1), 28–32.—The use of Zn acetate for the pptn. of S²⁻ was studied with reference to the co-pptn. of ZnSO₃. When the amount of Zn is >1·3 times that of the S²⁻, co-pptn. takes place; this is avoided in the presence of a small excess of Na₂CO₃, which reacts with the excess of Zn. The sample (25 ml) containing <30 g of NaOH per litre is treated with water (200 ml), Na₂CO₃ soln. (10%, 20 ml) and a small amount of Zn acetate soln. (10%), diluted to 500 ml and further treated with Zn acetate soln. until the mixture gives no reaction with Pb acetate paper. The soln. is filtered and the S²⁻ are determined by the TAPPI method. This method compares favourably with the TAPPI and the Kurtnäcker methods (*Z. anorg. Chem.*, 1927, **161**, 201), in which a ZnCO₃ suspension is used. K. SAITO

2336. A semi-micro method of high accuracy for the determination of sulphur in wool. B. J. Myers (New Zealand Wool Industries Res. Inst., 444 George St., Dunedin). *J. Text. Inst. Trans.*, 1959, **50** (8), T494–T503.—The S content of the sample (≃0·2 g) is determined by wet oxidation to H₂SO₄ by a HNO₃ - HClO₄ mixture, followed by pptn. as BaSO₄. Factors affecting the quant. recovery of BaSO₄ are discussed, *viz.*, the quantities of HNO₃ and HClO₄ in relation to the weight of wool; the rate of oxidation (governed by the volume of HNO₃ and the temp.); the use of the minimum amount of K₂Cr₂O₇ to catalyse the oxidation and detect the approach of the end-point by a colour change from green to orange; the acidity of the solution to be precipitated; the amount of BaCl₂ used and method of adding it; the time allowed for the aggregation of the precipitate before filtration. A range of 3·21 to 3·32% of S in a Merino top wool was determined with a standard deviation of 0·026% (61 determinations) by the procedure recommended.

O. M. WHITTON

2337. Separation of mixed polymers by a chromatographic method. W. J. Langford and D. J. Vaughan (Production Lab., Tufnol Ltd., Perry Barr, Birmingham, England). *J. Chromatography*, 1959, **2** (5), 564–565 (in English).—To separate poly(vinyl acetate), poly(vinyl butyral), cellulose acetate, polystyrene and poly(vinyl chloride) by two-dimensional chromatography on paper, develop first with *iso*butyl methyl ketone and second with methanol - *iso*butyl methyl ketone.

A. R. ROGERS

2338. Analytical studies on surface-active substances. II. Conjugated polyoxyethylene glycols. C. Jacobelli Turi (Ist. Sup. Sanità, Roma, Italy). *R. C. Ist. Sup. Sanit.*, 1959, **22** (7), 727–746.— "Conjugated" surface-active substances, *i.e.*, those in which the polyoxyethylene chain is linked to other substances, are classified, on the basis of their behaviour on saponification and the presence or absence of N or S in the molecule, into 6 groups, *viz.*, ethers of primary aliphatic alcohols, phenolic ethers, esters, amides, amines and sulphides. A systematic scheme is described for the identification of the individual members of the groups.

L. ZANONI

2339. Polarographic determination of residual acrylonitrile [vinyl cyanide] in polymeric systems. G. C. Claver and M. E. Murphy (Plastics Div., Monsanto Chemical Co., Springfield, Mass., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1682–1683.—To determine residual vinyl cyanide monomer in styrene - vinyl cyanide copolymer, dissolve the sample in a $0.1\,M$ soln. of tetrabutylammonium iodide in dimethylformamide containing 5% of H_2O, pass N for 20 min. and measure the polarogram from -1.3 to -2.3 V *vs.* the mercury-pool anode. The concn. of vinyl cyanide monomer in the range 30 to 200 p.p.m. is determined with a relative accuracy of $\pm3.6\%$. The presence of styrene monomer causes no interference.   A. R. ROGERS

2340. Determination of vinylpyridine, acrylonitrile [vinyl cyanide] and acrylic acid units in elastomeric polymers. J. E. Burleigh, O. F. McKinney and M. G. Barker (Res. Div., Phillips Petroleum Co., Bartlesville, Okla., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1684–1686.—Standard Kjeldahl and non-aqueous titration procedures have been modified for use in the analysis of acid and base elastomers. A mixture of K_2SO_4, Se and HgO $(30:1:2)$ is used as catalyst with a digestion time of $\simeq 6$ hr. in the Kjeldahl determination of N in polymers containing pyridyl groups; digestion for 1 to 2 hr. is adequate for polymers containing vinyl cyanide or carbamate groups. Basic nitrogen in butadiene - vinylpyridine copolymers is titrated with $HClO_4$ in glacial acetic acid, with benzene, bromobenzene or chlorobenzene as solvent; acetone is added if necessary to solubilise any ppt. that forms. Acrylic acid units in copolymers with butadiene are titrated with Na methoxide and benzene; the solvent is benzene or tetrahydrofuran, pyridine or an ethanol - toluene mixture, according to the content of acrylic acid.

A. R. ROGERS

2341. Analysis of acrylonitrile [vinyl cyanide] - methylvinylpyridine copolymers. C. Stafford, jun., and P. E. Toren (Phillips Petroleum Co., Bartlesville, Okla., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1687–1689.—To determine vinyl cyanide (**I**) in copolymers of **I** and 2-methyl-5-vinylpyridine (**II**) and in partially hydrolysed copolymers, heat the sample under reflux with 54% H_2SO_4 for 2 hr., make alkaline and submit to a Kjeldahl-type NH_3 distillation and titration. To determine **II**, heat the sample under reflux with 54% H_2SO_4 for 45 min., dilute the product with $36\,N\ H_2SO_4$, and measure the extinction at 270 mμ with correction for irrelevant absorption estimated from readings at 235 mμ and 295 mμ. The confidence limits $(P = 0.95)$ for the mean of duplicate determinations are $\pm0.6\%$ absolute for **I** and $\pm1.6\%$ relative for **II**.   A. R. ROGERS

2342. Characterisation of saturated polyesters by differential thermal analysis. D. A. Anderson and E. S. Freeman (Pyrotechnics Chem. Res. Lab., Picatinny Arsenal, Dover, N.J., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1697–1700.—Differential thermal analyses have been carried out on an epoxy resin and 32 synthetic polymeric systems. The thermograms are unique for each system, and for comparisons between systems the endothermal bands between 250° and 450° can be used. The parameters used for this purpose are the areas of the endotherms, temp. at the peak, the number of peaks in a band, maximum rates of change in differential temp., and the temp. at which these maxima occur.

K. A. PROCTOR

2343. [Qualitative] infra-red spectroscopic analysis of polyester resins. R. J. Grisenthwaite (Res. & Devel. Dept., Bakelite Ltd.). *Brit. Plast.*, 1959, **32** (9), 428–429, 439.—The absorption bands of soln. of resins in styrene over the 2 to 15-μ region are described, together with their use for identifying fumaric, phthalic, adipic, chlorendic (hexachloro-tetrahydro*endo*methylenephthalic), carbic (tetrahydro*endo*methylenephthalic), *iso*phthalic, terephthalic and tetrachlorophthalic acids, propane-1:2-diol, ethanediol, diethylene glycol, triallyl cyanurate, methyl methacrylate and tri-(chloroethyl) phosphate.   S.C.I. ABSTR.

2344. Determination of hydroxyl numbers by near-infra-red absorption. C. L. Hinton (Res. Center, U.S. Rubber Co., Wayne, N.J., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1610–1612.—Duplicate determinations of hydroxyl numbers can be performed in about 30 min. with a Beckman Model DK-2 spectrophotometer. The results obtained on several polyesters and polyethers show an average difference of $<1\%$ between the chemical and near-infra-red methods. Amides, amines and oximes, besides hydroxyl-containing compounds such as alcohols, absorb in the 2.6 to 3.2-μ region used for the analysis, but any interference from such compounds can be corrected provided that the qualitative composition of the polyester or ether is known.

K. A. PROCTOR

2345. Quantitative determination of the composition of the copolymer of tetrafluoroethylene with trifluoroethylene by infra-red spectroscopy. L. I. Tarutina (Sci.-Res. Inst. of Polymerisation Plastics, Leningrad). *Zhur. Anal. Khim.*, 1959, **14** (4), 504–505.—The method is based on the measurement of the trifluoroethylene content of the copolymer by means of the absorption at 2987 cm^{-1} (with a LiF prism) due to the C–H group. A standard curve of extinction D *vs.* film thickness d_1 (microns) is prepared from polytrifluoroethylene, the extinction of a film of thickness d of the copolymer is measured at 2987 cm^{-1}, and the effective thickness d_1 of the trifluoroethylene contained in the copolymer film is read off from the calibration curve. The ratio of the effective thicknesses of trifluoroethylene and tetrafluoroethylene is then $d_1/(d-d_1)$ which, to obtain the ratio of the numbers of the molecules of the two components, must be multiplied by $K = (\rho_1/\rho_2)\ (M_2/M_1)$, where ρ_1, ρ_2, M_1 and M_2 are the densities and mol. wt. of tri- and tetra-fluoroethylene, respectively. For these compounds K is close to unity, so that the trifluoroethylene content of the copolymer is $(d_1/d)100\ \%$. The implied assumptions of this method, that the volume of the copolymer is an additive function of the volumes of its components and that the absorption of a C–H

group is identical in trifluoroethylene and in its copolymer, are shown to be correct by analysing standard copolymers, whose composition is known from an analysis of the gaseous mixture of monomers at 750 cm^{-1} (with a NaCl prism) for trifluoroethylene, which alone of the two components absorbs in this region.   C. D. KOPKIN

2346. Lead tetra-acetate method of determining small amounts of water in polyorganosiloxane liquids. Ya. I. Chulkov (All-Union Electro-tech. Inst.). *Zavod. Lab.*, 1959, **25** (8), 928–930.—The method is based on the quant. reaction of Pb tetra-acetate with H_2O to give PbO_2 and acetic acid. It is suitable for the determination of H_2O in various organic liquids, *e.g.*, benzene, diethyl ether, pyridine, acetone, and, in particular, liquids of the composition $[-(C_2H_5)_2SiO-]_n$, where n is between 3 and 12. The sample (5 to 10 g), together with 15 ml of a reagent soln. (prepared by dissolving 3 g of Pb tetra-acetate in 100 ml of anhydrous benzene and separating the clear soln. from any ppt.), is introduced into a glass ampoule which is then sealed and heated at 65° for $<$ 30 min. The ampoule is opened, then sealed with a rubber bung and centrifuged. The liquid is removed through a capillary tube and the tube is washed with anhydrous benzene. The PbO_2 is dissolved in 20 ml of a soln. prepared by adding 50 ml of 36·4% NaOH soln. to 50 ml of cooled glacial acetic acid, then testing with litmus and adding Na_2CO_3 if the reaction is acid, and finally adding a soln. of 7·5 g of KI in 50 ml of water. The liberated iodine is titrated with 0·02 N $Na_2S_2O_3$ in the presence of starch. Results are satisfactory with samples containing from 1 to 20 mg of H_2O in 0·5 to 10 ml of sample.   G. S. SMITH

2347. Use of gas chromatography in the plastics industry. J. Haslam (I.C.I. Plastics Div., Welwyn Garden City, Herts.). *Chem. Age*, 1959, **82**, 169–170.—Applications in the industry, especially to the determination of small amounts of impurities in monomers and to the identification of the depolymerisation products of polymers or copolymers, are discussed.   N. E.

2348. Determination of total sulphur in vulcanised rubber by the perchloric acid method. Fumio Fujita, Hideo Matsushita and Hiroshi Omori (Res. Inst., Toyo Rubber Ind., Ibaraki-shi, Osaka). *J. Soc. Rubb. Ind., Japan*, 1959, **32** (4), 260–263.—The $HClO_4$ - HNO_3 - Br method of Mackay (*J. Soc. Chem. Ind.*, 1930, **49**, 233T) was modified to determine the $SO_4{}^{2-}$ gravimetrically. The removal of $ClO_4{}^-$ with K^+ is not necessary and the time taken for a determination is $\simeq$ 8 hr. *Procedure*—Decompose the sample (0·5 g) with HNO_3 (sp. gr. 1·38) (10 ml) satd. with Br and 70% $HClO_4$ (5 ml) on a water bath for 30 min. Heat to white fumes, cool, add 5 N HCl (5 ml) and filter. Dilute the filtrate to 300 ml, add a slight excess of 10% $BaCl_2$ soln. and filter off and weigh the $BaSO_4$.   K. SAITO

2349. Polarographic determination of accelerators in rubber and auxiliaries. O. Rozací (Forschungsinst. f. Gummi u. Plast-Technol., Gottwaldov, Czechoslovakia). *Plaste u. Kautsch.*, 1959, **6** (8), 386–389.—Procedures have been developed for the polarographic determination of the following groups of rubber accelerators—thiazoles [*e.g.*, mercaptobenzothiazole (**I**) or dibenzothiazolyl disulphide (**II**)]; thiurams (*e.g.*, tetramethylthiuram disulphide); and thiocarbamates (*e.g.*, Zn diethyldithiocarba-

mate). Anodic or cathodic polarisation, and acid or alkaline media, are used as appropriate. **I** and **II** may be separated by their different solubilities in ethanol, and the concentration of either determined when it is present as an impurity in the other. The accelerators may be determined in rubber after extraction with acetone or other suitable solvent, and in this way changes in concentration during the vulcanisation process may be followed.   L. A. O'NEILL

2350. Quantitative determination of zinc diethyldithiocarbamate. J. W. H. Zijp (Rubb. Res. Inst. T. N. O., Delft, Holland). *I.R.I. Proc.*, 1959, **6** (4), 108–111.—A detailed study is presented of the conditions for the photometric determination in rubber compounds of zinc diethyldithiocarbamate accelerator (**I**) as the brown copper complex (**II**). *Procedure*—The finely divided rubber vulcanisate is extracted with benzene for 8 hr. Uncured test samples are extracted with benzene - methanol (1 : 1). The extract is partially evaporated, transferred to a 10-ml flask, 2 ml of benzene containing 40 mg of Cu oleate is added and the mixture is diluted to volume with benzene. On a strip of completely acetylated filter-paper (Whatman No. 1, 13 cm $\times$ 52 cm) is placed an appropriate amount of the soln.; after drying the spot and developing the chromatogram for 16 hr. by the ascending method with ethanol - benzene (1 : 1) as the mobile phase, the paper strip is dried, the brown spot of **II** is cut out and eluted with benzene for 90 min. The soln. is then diluted to 25 ml and the extinction is measured at 430 mμ against benzene. The content of **I** is found from a calibration curve which is constructed by means of pure **II**. It is suggested that all operations should be carried out in darkness or subdued daylight. This method gives satisfactory results and is not adversely affected by accelerators and antioxidants that do not react with Cu oleate; **I** cannot be determined in the presence of thiuram disulphide, which forms diethyldithiocarbamate during vulcanisation and extraction. Other dialkyldithiocarbamates may also be determined by this method.   B. B. BAUMINGER

2351. Analysis of a rubber mixture. V. A. Cirillo (Atlantic Refining Co., Philadelphia, Pa.). *Anal. Chem.*, 1960, **32** (2), 299.—Infra-red data are given for neoprene, butyl, natural and GR-S rubbers.   N. E.

2352. Terpolymer rubbers. Standardisation of infra-red analysis by chemical and radio-tracer methods. G. B. Sterling, J. G. Cobler, D. S. Erley and F. A. Blanchard (The Dow Chemical Co., Midland, Mich., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1612–1615.—The i.r. spectroscopic measurements of a methyl *iso*propenyl ketone - butadiene - vinyl cyanide terpolymer have been standardised by employing a combination of radioactive-tracer techniques and chemical methods. This eliminates the need for standards of known composition for i.r. analysis, and is generally applicable when a component of the polymer can be synthesised from radioactive materials.   K. A. PROCTOR

2353. Infra-red method for determining small amounts of dipentaerythritol hexanitrate in PETN [pentaerythritol tetranitrate]. W. E. Fredericks and F. Pristera (Picatinny Arsenal, Feltman Res. and Engng Labs., Dover, N.J.). *U.S. Atomic Energy Comm.*, Rep. PA-TN-30, April, 1959. 5 pp.—The method involves the preparation of a 15% soln. of

the sample in methyl cyanide, measuring the extinction of this soln. at the peak of the 8·9-μ band in a 1·0-mm rock-salt cell, and instrumental compensation of the cell, solvent and pentaerythritol tetranitrate (**I**). The net extinction was converted to percentage dipentaerythritol hexanitrate (**II**) by means of a previously established relationship between extinction and concn. This relationship has been established with standards. The developed method is applicable to the determination of as little as a few tenths of 1% of **II** in **I** with a precision of ≃0·1%.   NUCL. SCI. ABSTR.

See also Abstracts—**2032**, Carbon in organic compounds. **2033**, Organic micro-analysis. **2035**, Improved phenylhydrazine reagent for sugars. **2158**, Nitrogen in nitrocellulose. **2159**, Separation of NF₃ from CF₄. **2491**, Benzene in air. **2535**, Characterisation of organic compounds by gas chromatography.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Biological fluids, animal and vegetable tissues

2354. Estimation of total body water using a colorimetric method for the determination of antipyrin [phenazone] in plasma. D. Mendelsohn and N. W. Levin (Witwatersrand Univ., Johannesburg, S. Africa). *J. Lab. Clin. Med.*, 1959, **54** (2), 311–319.—In the proposed method phenazone (**I**) in acetic acid is treated with NaNO₂ and, after removal of the excess of NO₂⁻, the resulting nitroso compound is coupled with 1-naphthylamine and the extinction is measured at 580 mμ. The calibration curve is rectilinear up to 50 μg of **I**. The method requires 2 ml of plasma and its application to the determination of total body water is discussed.
W. H. C. SHAW

2355. Method for continuous polarographic determination of oxygen in blood *in vivo*. S. Österlind (Företagens Hälsokontroll, Stockholm, Sweden). *J. Polarographic Soc.*, 1959, (2), 28–29.—In experiments with rabbits, blood was led continuously through a small dropping mercury electrode vessel by inserting two thin cannulae into the carotid artery. The O content of the blood was followed, as a function of time, from the changes in the diffusion current at a constant p.d. of 1 V. Experiments have also been made with a needle having a centrally placed rotating platinum electrode to eliminate the use of mercury; the skin electrode used in cardiography can possibly be used to replace the mercury anode. The method is very sensitive in indicating changes in content of O in circulating arterial and venous blood.   H. F. W. KIRKPATRICK

2356. Improved flame-spectrophotometric serum calcium analyses by the addition of EDTA. R. Herrmann and W. Rick (Hautklin. u. Med. Klin. d. Justus-Liebig-Univ., Giessen, Germany). *Naturwissenschaften*, 1959, **46** (16), 492.—Dilution of serum (1:20) with a 5% aq. soln. of the ammonium salt of EDTA instead of with H₂O eliminates errors due to protein binding and other variable factors, *e.g.*, P content.
H. F. W. KIRKPATRICK

2357. Determination of magnesium in normal and pathological blood. P. Jannella (Ist. Chim. Biol., Univ., Naples). *Biochim. Appl.*, 1959, **6** (4), 187–190.—The method is based on the colorimetric determination of P by the method of Fiske and Subbarow (*J. Biol. Chem.*, 1925, **66**, 375). The serum or plasma is treated with trichloroacetic acid, and centrifuged, and Ca is removed from the clear supernatant liquid by pptn. with ammonium oxalate. Magnesium is pptd. with 2% KH₂PO₄ soln. and aq. NH₃, and the P in the ppt. is determined. Results are quoted for normal and various pathological cases, and recoveries of Mg quoted are from 92 to 98%.   E. C. APLING

2358. Micro-determination of phosphoric acid by solvent extraction. Atsunobu Yoda (Fac. of Sci., Kobe Univ., Higashinada-ku, Kobe). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (5), 488–490.—The extraction method of Wadelin and Mellon (*cf. Anal. Abstr.*, 1954, **1**, 679) was modified for the determination of <0·3 μg of P in biological materials. Butyl acetate (**I**) extracts the least amount of the excess of MoO₄²⁻, and separates well from the aq. layer. For deproteinisation, HClO₄ is the best reagent (Allen, *Biochem. J.*, 1940, **34**, 858). The calibration curve is linear for 0·5 to 5 μg of P per 5 ml. The deproteinised sample is mixed with HClO₄ (0·5 ml) and (NH₄)₂MoO₄ soln. (5%, 2 ml) and shaken with **I** (5 ml); it is then centrifuged (1000 r.p.m., 3 to 5 min.) and the extinction of the **I** layer is measured at 310 mμ.
K. SAITO

2359. Colorimetric micro-determination of antimony in blood and urine with crystal violet. Yu-Cheng Leu (Dept. of Chem., Chungking Med. Coll.). *Acta Pharm. Sinica*, 1959, **7** (5), 171–174.—The sample (1 to 4 ml of blood or 0·5 to 5 ml of urine) is digested with a mixture of HClO₄, HNO₃ and H₂SO₄; after complete decomposition of the HClO₄ the mixture is cooled in ice, 3 ml of aq. NaCl soln. is added slowly, followed by 3 ml of water, and the HSbCl₆ is extracted with *iso*amyl acetate (5 ml). The acetate layer is washed with 6 N HCl (5 ml) and then treated with a mixture of 0·1% aq. crystal violet soln. (0·5 ml) and 0·3 N HCl (5 ml). The colour intensity of the amyl acetate layer is measured against a blank at 590 mμ in 1-cm cells. The colour increases with the amount of crystal violet, but decreases with that of HCl. The method proposed is more sensitive than that with Rhodamine B (C.I. Basic Violet 10). It gives a low blank and is not seriously affected by Fe. Beer's law is obeyed over the range 0 to 2·5 μg of Sb. Recovery tests show that the error is ±5% for 1 μg of Sb and ±10% for 0·1 μg.   S. H. YUEN

2360. Spectrophotometric determination of chloride in urine. M. Itano, L. A. Williams and B. Zak (Dept. of Path., Wayne State Univ. Coll. of Med., Detroit, Mich., U.S.A.). *Amer. J. Clin. Path.*, 1959, **32** (3), 213–215.—The method is based on the reaction of Cl⁻ with mercuric chloranilate (**I**) to produce HgCl₂ and purple chloranilic acid. *Procedure*—The sample (0·2 ml) is mixed with 1% HNO₃ soln. (10 ml) and 20 to 25 mg of **I** is added. The mixture is shaken well and centrifuged. The extinction of the supernatant liquid is measured at 540 mμ. The technique is precise and accurate.
P. NICHOLLS

2361. Determination of iron in urine. C. Vecchione, S. Fati and P. Piccoli (Ist. Med. del Lavoro, Univ., Naples). *Biochim. Appl.*, 1959, **6**

(5), 257–264.—A modified wet-ashing procedure is proposed for the determination of Fe by colorimetry with 1:10-phenanthroline. *Procedure*—Evaporate the sample (50 ml) to dryness on a sand bath three times with HNO_3 (5 ml, 1 ml, 1 ml). Dissolve the residue in H_2SO_4 (1 ml), decolorise the soln. with 30% H_2O_2 soln. (1 ml) and evaporate to fumes. Reduce with 2% $Na_2S_2O_4$ soln. (2 ml), evaporate to fumes, and cool. Dissolve the residue in water (5 ml) and determine the Fe in a 2-ml aliquot by the 1:10-phenanthroline method. Results obtained show the method to have improved reproducibility and sensitivity compared with the H_2SO_4 digestion procedure of Hemmeler ("*Métabolism du fer*," Masson et Cie, Paris, 1951).

E. C. APLING

2362. Determination of iron in urine using 4:7-diphenyl-1:10-phenanthroline [bathophenanthroline]. P. Collins and H. Diehl (Dept. Chem., Iowa State Univ., Ames, U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1692–1693.—A 50-ml aliquot of urine is wet-ashed with 25 ml of HNO_3 and 10 ml of 70% $HClO_4$. The hot digest is treated with 2 ml of 10% hydroxyammonium chloride and 5 ml of $0.001 M$ 4:7-diphenyl-1:10-phenanthroline (**I**). The pH is adusted to 5 and the Fe^{II} - **I** complex is extracted successively with 4-, 2- and 2-ml portions of nitrobenzene. The final volume is adjusted to 10 ml with ethanol, and the extinction is read at 538 mμ.   S. BAAR

2363. Activation analysis of trace cobalt in tissue using 10·5-minute cobalt-60m. D. G. Kaiser and W. W. Meinke (Univ. Michigan, Ann Arbor). *U.S. Atomic Energy Comm.*, Rep. AECU-4289, 1959, 13 pp.—Microgram amounts of Co from vitamin B_{12} were determined by rapid activation analysis in rat-kidney tissue and in vitamin preparations. A 15-min. radiochemical separation procedure coupled with γ-ray spectrometry made possible the use of the 10·5-min. ^{60m}Co radio-isotope. With this procedure the lower limit of detection at a thermal neutron flux of 10^{13} per sq. cm per sec. was $\simeq 5 \times 10^{-8}$ g of Co.   NUCL. SCI. ABSTR.

2364. Determination of urinary phenol as an index of exposure to benzene. A. G. Dettori and A. Maggi (Clin. Med. Generale, Univ. Parma, Italy). *Diagn., Lab. e Clin.*, 1958, **14** (4), 150–158.—Urinary phenol was determined for 22 normal male subjects, 33 workers in 3 petroleum refineries, and 6 tanker drivers exposed to benzene, by the method of Porteous and William (*cf. Biochem. J.*, 1949, **44**, 46). Mean results for normal subjects were 0.61 ± 0.33 mg of total phenols per 100 ml. and for the 39 exposed subjects were 2.53 ± 1.74 mg per 100 ml. In a few cases, particularly those subjects exposed to benzene intoxication, free urinary phenol was found. The differences between normal subjects and petroleum workers, and between factory groups, were statistically highly significant and were apparently correlated with the degree of exposure to intoxication by benzene.

E. C. APLING

2365. Determination of sulphisoxazole [sulphafurazole] and sulphadimethoxine in blood and plasma. H. L. Rosenthal and L. Jud (Rochester Gen. Hosp., N.Y., U.S.A.). *J. Lab. Clin. Med.*, 1959, **54** (3), 461–466.—The compounds are determined in trichloroacetic acid extracts of blood and serum by the method of Bratton and Marshall (*J. Biol. Chem.*, 1939, **128**, 537). It is shown that both compounds

are adsorbed on pptd. blood proteins but much less on pptd. serum proteins, and that accurate results can be obtained on serum after the application of a correction factor for loss by adsorption.

W. H. C. SHAW

2366. Determination of bromosulphonephthalein in serum by means of acetone precipitation of proteins. R. J. Henry, N. Chiamori and A. G. Ware (Bio-Science Res. Found., Los Angeles, Calif., U.S.A.). *Amer. J. Clin. Path.*, 1959, **32** (2), 201–203.—Acetone (80% by vol.) (8 ml) acidified with acetic acid is added to 2 ml of serum. The mixture is shaken and centrifuged and the extinction of the supernatant liquid is measured at 585 mμ. The extinction is measured again after the addition of 2 drops of 10% NaOH soln. The difference in the two readings is proportional to the concentration of bromosulphonephthalein. The mean recovery by this method is 97·3%.   P. NICHOLLS

2367. Determination of glycerol in blood - glycerol mixtures. C. A. Crutchfield and H. A. Sloviter (Pennsylvania Univ. Med. Sch., Philadelphia, U.S.A.). *J. Lab. Clin. Med.*, 1959, **54** (3), 478–483.—This is an adaptation of the periodate oxidation method for glycerol, in which interference by glucose is minimised by preliminary destruction with hot aq. NaOH soln. on a portion of the filtrate from the sample treated with trichloroacetic acid. From titrations with and without alkali treatment, the glycerol content can be determined with a mean error of $\pm 1.4\%$, while a less precise estimate of the glucose content can also be made. The method is considered to be of general application to mixtures of glycols and reducing sugars.

W. H. C. SHAW

2368. Separation of galactose, glucose and lactose in urine by paper chromatography. A. J. Philippu (Lab. of Physiol., Univ. of Athens, Greece). *Anal. Chem.*, 1959, **31** (10), 1743–1744.—A wedge-shaped Whatman No. 1 filter-paper (38 cm $\times$ 4 cm) is used. The sample (0·001 ml) is applied at the tail end (0·4 cm wide) and developed with the solvent system *n*-propanol - ethyl acetate - H_2O - α-picoline (14:2:4:9) for 36 hr. The strip is sprayed with aniline - phosphoric acid in *n*-butanol (*cf.* Bryson and Mitchell, *Nature*, 1951, **167**, 164) then heated at 105° for 3 to 5 min. The method is sensitive to 5 μg of each sugar.   S. BAAR

2369. Quantitative determination of reducing sugars after separation by paper chromatography. A. J. Philippu (Lab. of Physiology, Univ. of Athens, Greece). *Anal. Chem.*, 1959, **31** (10), 1615–1616.—Extract the sugars from the paper with methanol (10 ml) at 65° for 20 min. Add a mixture of *n*-propanol, aniline and orthophosphoric acid (150:1:1) (3 ml), heat in a bath of boiling water for 20 min., dilute with methanol (5 ml) and measure the extinction at 400 mμ. Perform a blank determination on sugar-free paper dipped in methanol. Quantities of glucose, fructose, lactose and galactose from 50 to 500 μg can be determined with a standard deviation of <5 μg.

A. R. ROGERS

2370. Micro-determination of reducing sugars in blood. Michio Ui (Pharm. Inst., Med. Sch., Univ. Hokkaido, Japan). *Chem. & Pharm. Bull., Tokyo,* 1959, **7** (6), 695–698 (in English).—Blood (0·02 ml) and water (1·78 ml) are placed in a tube, 20% trichloroacetic acid (0·2 ml) is added to precipitate

the proteins, and the mixture is centrifuged. To the supernatant liquid (0·5 ml) in a test-tube is added a 1:1 mixture of 0·1% $K_2Fe(CN)_6$ soln. and 2·1% Na_2CO_3 soln. in 0·25 N NaOH (1·0 ml). After heating the mixture in a boiling-water bath for 15 min. and cooling for 3 min., 0·1% $(NH_4)_2SO_4.Fe_2(SO_4)_3.24H_2O$ soln. in 0·3 N H_2SO_4 (1·0 ml) is added. The vol. is made up to 6·0 ml with water and the extinction is read immediately at 660 mμ. As little as 2 μg of glucose may be determined by this method.

R. A. BRENAN

2371. Separate determination of glucuronic acid and glucuronides in biological fluids by ion-exchange resin. Morizo Ishidate and Mitsuo Watanabe (Fac. of Pharm. Sci., Univ. Tokyo). *Chem. & Pharm. Bull., Tokyo,* 1959, **7** (6), 685–689 (in English).—The sample, made slightly alkaline with NaOH, is passed through a column of Amberlite IRA-411. After washing the column with water to desorb sugars and impurities, the free glucuronic acid is eluted with 0·25 N NH_4Cl (adjusted to pH 8 with aq. NH_3), the N-glucuronides are then eluted with N HCl, and finally the O-glucuronides are eluted with methanolic HCl (44 ml of conc. HCl diluted to 500 ml with methanol). The separated fractions are determined colorimetrically at 520 mμ after reaction with carbazole and H_2SO_4.

R. A. BRENAN

2372. Simple method for quantitative analysis of sugars by ion-exchange chromatography. M. Nakamura and K. Mori (Tokyo Univ., Japan). *Biochim. Biophys. Acta,* 1959, **34** (2), 546–548.—Chromatography is carried out on Dowex 1-X8 ion-exchange resin (borate form) from which the sugars are eluted with 0·001 to 0·02 M $Na_2B_4O_7$ in 50% aq. ethanol. A simplified procedure for preparing extracts of vegetable tissues is described.

W. H. C. SHAW

(See also *Anal. Abstr.,* 1960, **7**, 2373.)

2373. Quantitative analysis of sugars in plant extracts by ion-exchange chromatography with special reference to the examination of conditions for preparing the sample sugar solutions. Kenji Mori and Michinori Nakamura (Dept. of Agric. Chem., Univ. Tokyo, Bunkyo-Kan). *Bull. Agric. Chem. Soc. Japan,* 1959, **23** (5), 389–397 (in English).—Conditions governing the quant. recovery of sugars from plant extracts by ion-exchange chromatography of the borate complexes (Khym and Zill, *J. Amer. Chem. Soc.,* 1952, **74**, 2090) are investigated. Losses occur during the removal of non-sugar components by pptn. with Pb acetate and by adsorption of sugars by ion-exchange resins. A satisfactory method for determining sugar in potato tubers is described. *Procedure*—Mash the sample of peeled potatoes (50 g) with 95% ethanol (200 ml) containing a small amount of $CaCO_3$ and heat under reflux for 1 hr. Extract with hot 80% ethanol (4 × 70 ml), combine the extracts and record the vol. Concentrate an aliquot *in vacuo* to ≃10 ml at a temp ≯40° and transfer it to a centrifuge tube with the aid of a small vol. of water; add 30% Pb acetate soln. (0·4 ml); centrifuge, decant off the supernatant liquid and wash the ppt. thrice with water; to the combined supernatant liquid and washings add 3 ml each of Amberlite IR-120 and IR-4B resins, stir for 20 min. and filter; wash the resins and combine the filtrate and washings, concentrate *in vacuo*, add sufficient 0·005 M $Na_2B_4O_7$ to give a final concn. of 0·001 M,

and determine the sugars by the method of Khym and Zill. Results for potatoes stored at 6° and 30° are discussed.   H. B. HEATH

2374. Studies on the chemistry and physiology of some metabolically important acids. III. A simple paper-chromatographic method for the detection of *iso*citric acid in plant materials. R. Pohloudek-Fabini, C. Wollmann and H. Wollmann (Pharm.-chem. Inst. und Zentrale Univ.-apotheke der Univ., Greifswald, Germany). *J. Chromatography,* 1959, **2** (5), 525–530 (in German).—Separate *iso*citric acid (**I**) from citric acid (**II**) and other acids by descending chromatography on S. & S. 2043b filter-paper with a H_2O-saturated mixture of *iso*-amyl alcohol, $CHCl_3$ and 85% formic acid (4:1:1) as solvent. The R_F values are 0·12 and 0·14 for **I** and **II**, respectively, and the solvent should be allowed to flow off the paper to ensure adequate separation. Allow the chromatogram to dry over-night at room temp., spray with Schweppe's reagent [mix a soln. of glucose (2 g) in H_2O (20 ml) with aniline (2 ml) and ethanol (20 ml) and dilute to 100 ml with *n*-butanol] and dry at 125°; the acids show as brown spots on a white background. **I** has been identified in various plant extracts by this method.   A. R. ROGERS

2375. Determination of hydroxyl groups in alkali lignin by methylation with dimethyl sulphate. J. Farkaš (Res. Inst. Paper and Cellulose, Bratislava, Czechoslovakia). *Chem. Zvesti,* 1959, **13** (9), 536–540.—By studying the influence of time and temp., conditions were established for the determination of hydroxyl groups in alkali lignin, based on methylation with dimethyl sulphate (**I**) at 60°. *Procedure*—Dissolve lignin (9 g) in 10% NaOH soln. (45 ml) in a Walter flask. Add **I** (10·5 ml) drop-wise with cooling, completing the addition within 30 min. Set aside at 60° for 6 hr. with stirring, add a small vol. of 25% H_2SO_4 soln., dilute with H_2O, filter off the product, wash it with H_2O and dry at 60°. Repeat the methylation twice more, and determine the methoxyl groups in the final product by a classical method.   J. ZÝKA

2376. Detection of flavanones and hydroxy-flavanones by a specific reaction. R. Neu (Chem. Forschungslab. d. Fa. Dr. Willmar Schwabe, Karlsruhe-Durlach). *Arch. Pharm., Berlin,* 1959, **292** (8–9), 431–437.—Flavanones and hydroxy-flavanones can be differentiated from flavones and hydroxyflavones by paper chromatography, since they give blue or reddish-violet spots with a 0·1% soln. of Rhodamine B (C.I. Basic Violet 10) in 4% HCl, whereas flavones and hydroxyflavones do not. As a confirmatory test, this chromatogram is moistened with 25% aq. NH_3, when the flavones and hydroxyflavones appear as yellow and brown spots. The individual compounds are identified by their R_F values in various solvents. The test can also be performed in a tube, the test soln., after the addition of Rhodamine B, being examined in u.v. light. Flavones and hydroxyflavones extinguish the fluorescence, whereas flavanones and hydroxyflavanones do not. R_F values are tabulated for 15 compounds and 15 vegetable drugs.   M. H. SAWISTOWSKA

2377. Quantitative paper chromatography of caro-tenoids. A. Jensen and S. L. Jensen (Norwegian Inst. of Seaweed Res., Trondheim, Norway). *Acta Chem. Scand.,* 1959, **13** (9), 1863–1868 (in English).—

By using circular chromatography on paper containing 20% of kieselguhr as filler, with acetone - light petroleum as solvent, 10 to 100-μg amounts of carotenoids present in brown seaweed and bacteria were separated into carotenes, mono- and dihydroxyxanthophylls, or their aliphatic equivalents; further separations of these could then be made. The resolution and quant. determination of a mixture (50 μg) of stereoisomers was achieved; the eluted components were determined spectrophotometrically. By using light petroleum - isopropyl alcohol as solvent, the chloroplast pigments of algae and grass extracts could also be separated. The influence of structure on R_F values is discussed; these values were reproducible to ±0·01 unit.

P. D. PARR-RICHARD

2378. Radial chromatography of bile pigments. L. Nassi (Clin. Pediat., Univ., Firenze, Italy). *Diagn., Lab. e Clin.*, 1959, **15** (4), 145–162.—An improved technique for radial paper-chromatographic separations of diazotised bile pigments is described. Chromatograms were run on Whatman No. 1 or No. 3 paper (22 cm diam.), previously washed with diethyl ether, with ethyl methyl ketone - n-propionic acid - water (15:5:6) as mobile phase. Concentration of the sample for application to the chromatogram is accomplished by repeated immersion of the tip of a folded pyramid of filter-paper in the diazotised solution, with air-drying between immersions, until the colour is sufficiently concentrated in a small area at the tip of the pyramid. The sample and mobile phase are then applied to the centre of the paper by using the folded pyramid as a wick.   E. C. APLING

2379. Differential determination of serum bilirubin. J. E. Ridgway (Clatterbridge Hosp., Cheshire, England). *J. Med. Lab. Technol.*, 1959, **16** (3), 222–224.—A method for the differential determination of directly reacting conjugated bilirubin that avoids the turbidity due to protein pptn. in aq. soln. is described. Serum (0·5 ml) and Van den Bergh diazo reagent (0·5 ml) are placed in two centrifuge tubes ("total" and "direct"). After 5 min., SnCl₂ soln. (prepared by adding 2·0 g of SnCl₂.2H₂O to 100 ml of boiling 0·1 N HCl and covering immediately with a layer of toluene; stable for 10 days) (0·1 ml) is added to the "direct" tube to destroy any remaining diazonium chloride. After 1 min., 85% ethanol (3·5 ml) is added to each tube and after 30 min. the proteins are pptd. by the addition of satd. (NH₄)₂SO₄ soln. (0·5 ml) to each tube, followed by centrifugation and filtration through a 7-cm Whatman No. 1 paper. The extinctions of the filtrates are measured at 520 mμ and compared with a suitable standard (e.g., methyl red). The "indirect" non-conjugated bilirubin is obtained by difference.   R. A. BRENAN

2380. Methods for the analysis of lipids extracted from human arteries and other tissues. C. J. F. Böttcher, F. P. Woodford, E. Boelsma-van Houte and C. M. van Gent (Dept. of Phys. Chem., Univ., Leiden). *Rec. Trav. Chim. Pays-Bas*, 1959, **78** (9–10), 794–814 (in English).—The analytical procedure described can be applied to samples of 50 mg or less. Freeze-dried material is extracted with methanol - CHCl₃ (1:2) and non-lipid material removed by passage through a column of powdered cellulose. After evaporation of the percolate, the residue is dissolved in light petroleum (boiling-range 40° to 60°) and phospholipids are removed by dialysis through a rubber membrane. Fatty acids

in the dialysate are removed with aq. KOH soln. and the neutral dialysate is passed through a column of silica gel; sterol esters are eluted from the column with benzene - light petroleum (3:7) and other lipids with 5% methanol in diethyl ether. After saponification, the fatty acids are methylated and separated by gas chromatography; phospholipids are first transmethylated before saponification. Quinol is used as an antioxidant during certain parts of the analysis.   P. D. PARR-RICHARD

2381. A semi-micro method for the colorimetric determination of the phosphorus content of lipids. I. Korpaczy (Inst. für Ernährungs- und Verpflegungswissenschaft, Budapest). *Fette, Seif., Anstrichmitt.*, 1959, **61** (9), 748–750.—The method of Thaler (cf. Ibid., 1952, **54**, 763) is modified to give an increased sensitivity by extracting the molybdophosphoric acid with n-butanol in the presence of sodium sulphate. The modified method has a sensitivity of 1 p.p.m. of phosphorus.

W. T. CARTER

2382. Nephelometric detection of lipids in chromatographic column effluents. A. C. Arcus (Nutrition Res. Dept., Medical School, Dunedin, New Zealand). *Anal. Chem.*, 1959, **31** (10), 1618–1620.—Place a soln. of the sample in methanol (1 ml containing <1 mg of lipid) in a test-tube (6 in. × ⅝ in.) which is inclined at an angle of 30° to the horizontal and which dips into a bath of water at 50°. Rotate the tube at 300 r.p.m. for 1 min. and add H₂O (2 ml) by injection into the rotating soln. through the needle of a syringe during a further 2 min. Remove the tube, dilute with H₂O to 10 ml and mix again. After a further minute, measure the light-scattering power of the lipid suspension in a poly(methyl methacrylate) cell. As little as 2 μg of cholesterol can be detected and larger amounts can be determined semi-quantitatively. The method has been used to show the presence of at least 17 components in the unsaponifiable matter of cod-liver oil.

A. R. ROGERS

2383. Separation of sugars, amino acids and organic acids with the aid of ion-exchange resins. Kh. Dilov and D. Georgiev. *Compt. Rend. Acad. Bulg. Sci.*, 1959, **12** (3), 227–230.—The ion-exchange resins Wofatit KPS 200 (cationite) and Wofatit L 150 (anionite) are used. An aq. soln. of the compounds is passed through a column of the cationite on which the amino acids are retained. The organic acids are retained when the percolate from the first column is passed through a column of the anionite. The percolate from the second column contains the sugars. The amino acids are eluted from the cationite with 2 N HCl, and the organic acids are eluted from the anionite with 0·5 N NaOH. The separation of the three groups of compounds is checked by treating standard soln. containing glucose, fructose, glutamic acid, glycine, aspartic acid, tryptophan, valine, serine, phenylalanine, methionine, leucine, and oxalic, malic, citric, tartaric and succinic acids in the same way. The error is <3% for sugars and amino acids, and <2·4% for the organic acids.

ABSTR. BULG. SCI. LIT.

2384. Amino-sugar determination in the hydrolysates of animal, plant and bacterial material. B. N. Gladyshev (Inst. Biochem., Acad. Sci., Moscow). *Biokhimiya*, 1959, **24** (5), 789–793.—A procedure is described for the preparation of a hydrolysate for determination of the amino-sugar

concn. by the Elson and Morgan method (*Biochem. J.*, 1933, **27**, 1823). Determinations showed that 70 to 80% of added glucosamine was recovered.

K. R. C.

2385. Improved method for the detection of N-acetylamino sugars on paper chromatograms. M. R. J. Salton (Manchester Univ., England). *Biochim. Biophys. Acta*, 1959, **34** (2), 308-312.— Improved sensitivity is attained by spraying chromatograms with a mixture (1:1) of 95% ethanol and 0·05 M Na$_2$B$_4$O$_7$. The papers are suspended in steam for 10 min. and then sprayed with a reagent consisting of a 2% solution of p-dimethylaminobenzaldehyde in glacial acetic acid - n-butanol - conc. HCl (10:30:0·4). As little as 1 μg of material can be detected and, by extraction of the coloured complexes, the method can be made quantitative.

W. H. C. SHAW

2386. The N-acetylation and estimation of hexosamines. G. A. Levvy and A. McAllan (Rowett Res. Inst., Bucksburn, Aberdeenshire, Scotland). *Biochem. J.*, 1959, **73** (1), 127-132.—Glucosamine and galactosamine are determined by the method of Reissig *et al.* (*J. Biol. Chem.*, 1955, **217**, 959) after pre-treatment in aq. soln. with acetic anhydride to convert them into the N-acetyl derivatives. The borate buffer used for chromogen formation catalyses the N-acetylation of the amino sugars. The method is very sensitive, convenient and reproducible.

J. N. ASHLEY

2387. Separation and determination of glucosaminic acid by column chromatography. L. Pandolfo and G. Di Chiara (Ist. Chim. Biol., Univ., Palermo). *Biochim. Appl.*, 1959, **6** (5), 281-286.—Glucosamine (**I**) and glucosaminic acid (**II**) were separated on a column (22 cm × 0·7 cm) of Dowex 50-X8 (200 to 400 mesh), which had been previously washed with a large vol. of 0·4 N HCl, and finally with 0·3 N HCl (100 ml). The sample soln. (0·2 to 1·0 ml, containing from 50 to 500 μg of **I** and **II**) was applied to the column and eluted with 0·3 N HCl at a flow-rate of 0·2 ml per min. Fractions (2 ml) were collected, **II** being eluted in fractions 15 to 18 and **I** in fractions 20 to 22. The identity and purity of the fractions were checked by spectrophotometry and by paper chromatography. For the determination, the fractions were neutralised with N NaOH and heated for 20 min. at 80° with 10% aq. pyridine soln. (1 ml) and a 1% ethanolic soln. of ninhydrin (2 ml). After cooling the soln., the extinction was measured at 520 mμ, and compared with that of standards similarly treated. Column recoveries for pure soln. and for additions made to biological homogenates were 100%.

E. C. APLING

2388. The concentration of serotonin [5-hydroxytryptamine] in normal human serum as determined by an improved method. R. B. Davis (Minnesota Univ. Hosp., Minneapolis, U.S.A.). *J. Lab. Clin. Med.*, 1959, **54** (3), 344-351.—Low and variable results obtained previously for 5-hydroxytryptamine (**I**) in serum are attributed to destruction in the sample before assay; this is prevented in the method described by the addition of ascorbic acid (41 mg) to the fresh blood sample (7 ml). After pptn. of proteins with ZnSO$_4$ and NaOH, **I** is extracted with butanol from the supernatant liquid and, after further purification and transfer to aq. HCl, is determined in a spectrofluorimeter. The fluorescence is measured at 550 mμ with activation at 295 mμ. Satisfactory recovery of added **I** is attained. The mean concentration in normal serum was 0·21 μg per ml with a range of 0·10 to 0·32 μg.

W. H. C. SHAW

2389. Nucleic acids of the liver of rats; ion-exchange chromatography of the purine and pyrimidine bases. C. M. Caldarera, R. Infante and F. Sanguinetti (Ist. Chim. Biol., Univ., Bologna). *Biochim. Appl.*, 1959, **6** (4), 172-179.—The fraction of rat liver insoluble in trichloroacetic acid was hydrolysed by treatment with 72% HClO$_4$ for 1 hr. at 100°. The liberated bases were separated on a column of Dowex 50 (0·78 sq. cm × 50 cm). Elution was maintained at 0·35 to 0·40 ml per min., initially with 1·5 N HCl and then successively with 2·5 N, 4 N and 6 N HCl. The course of the elution was followed by measuring the extinction of each fraction of the eluate at 260 mμ. Complete resolution of the nitrogenous bases was attained, with recoveries of 93 to 98%.

E. C. APLING

2390. Ultra-violet fluorescence of guanine on paper chromatograms and in solution. I. Ya. Barskii (Botanic. Inst., Acad. Sci., Leningrad). *Biokhimya*, 1959, **24** (5), 823-825.—Owing to the considerable increase in the brightness of the fluorescence of many substances when they are adsorbed on to filter-paper, the fluorescence spectra of spots of various substances (not named) can be photographed directly from paper chromatograms placed between a quartz cover-glass and a quartz microscope-slide, if a u.v. microscope fitted with a Brumberg spectrographic attachment (*Dokl. Akad. Nauk*, 1949, **61**, 43) is used. The curves of energy distribution for the fluorescence spectra of guanine soln. (in a micro-cell) and of guanine adsorbed on to a chromatographic paper, of especially low fluorescing capacity, from HCl, phosphate buffer and NaOH soln. (pH 1, 3, 5, 7, 9 and 14) are compared. The positions of the maxima differ widely in alkaline and acid media.

K. R. C.

2391. Determination of some nitrogenous components of the urine in mixed excreta of poultry by means of paper chromatography and paper electrophoresis. J. Wagner and E. Franzen (Inst. Tierernährung, Martin Luther Univ., Halle-Wittenberg, Germany). *Arch. Tierernährung*, 1959, **9**, 11-22.—R_F values are given for creatine, creatinine, uric acid, urea, guanidine, sarcosine, guanine, xanthine and allantoin in 10 solvent mixtures and for the electrophoretic mobilities of creatine, creatinine, urea and uric acid at different pH values at 600 V. The nitrogen partition of the excreta of hens maintained on mixed diets was estimated.

NUTR. ABSTR. REV.

2392. Separation of amino acids by thin-layer chromatography. E. Mutschler and H. Rochelmeyer (Pharm. Inst., Univ., Mainz). *Arch. Pharm., Berlin*, 1959, **292** (8-9), 449-452.—A procedure is described for the separation of amino acids by two-dimensional thin-layer chromatography on plates of silica gel buffered with a mixture of KH$_2$PO$_4$ and Na$_2$HPO$_4$ (prep. described). Ethanol (70%), 96% ethanol - 25% aq. NH$_3$ (4:1), and ethanol - aq. NH$_3$ - water (7:1:2) were employed as solvents; R_F values are tabulated for 13 amino acids.

M. H. SAWISTOWSKA

2393. Two-way separation of amino acids and other ninhydrin-reacting substances by high-voltage electrophoresis followed by paper chromatography. M. L. Efron (Med. Unit, Univ. Coll. Hosp. Med.

Sch., London). *Biochem. J.*, 1959, **72** (4), 691–694.—
The method gives a more rapid separation than two-
way paper-chromatographic methods, and a better
separation of most of the amino acids is obtained.
It is particularly useful for plasma and cerebrospinal
fluid because they can be deproteinised in a few
minutes with picric acid and the tedious process of
ultra-filtration is avoided. Also, de-salting is not
needed, because it occurs automatically during the
electrophoresis. The usual 24-hr. first run in phenol
is replaced by high-voltage electorphoresis which
requires only 1·5 hr. The entire separation takes
only 9 to 10 hr. instead of the customary 48 hr.
The spots given by the amino acids after being
sprayed with ninhydrin are rounder and more
compact, and there is very little diffusion of the
spots. A diagram is given which shows the positions
taken up by 43 ninhydrin-reacting substances when
they are separated by this method.

J. N. ASHLEY

**2394. Microbiological assays [of amino acids] with
several strains of *Leuconostoc mesenteroides*.**
M. C. Shekleton and W. C. Haynes (Fermentation
Lab., N. Utilization Res. Dev. Div., U.S. Dept.
Agric., Peoria, Ill.). *J. Bacteriol.*, 1959, **77**, 114–
115.—Seven P-60 strains of *L. mesenteroides* were
tested for suitability for the routine assay of L-lysine,
L-valine and DL-*iso*leucine. Strain NRRL B-1153
consistently gave a more closely linear response
than did the other strains and is therefore recom-
mended for assay procedures.

NUTR. ABSTR. REV.

**2395. Influence of amino acids upon the anthrone
reaction of uronic acids.** J. R. Helbert and K. D.
Brown (Veterans Admin. Hosp., Downey, Ill.,
U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1700–1702.—
The influence of various amino acids on the intensity
of the colour produced by heating glucuronic acid,
galacturonic acid, mannurone or idurone with
0·160% (w/v) anthrone soln. in 27·5 N H_2SO_4 was
investigated. Tryptophan, cysteine, methionine,
phenylalanine, tyrosine, proline, hydroxyproline,
histidine, glutamic acid, lysine, valine and arginine
were tested, and, of these amino acids, only trypto-
phan produced a colour with anthrone alone. In
mixtures of tryptophan with glucuronic acid or
mannurone, the extinctions of the individual
components at 550 mμ are additive. Methionine
enhances the colour produced by all the uronic acids
and anthrone; cysteine may enhance or depress this
colour intensity. S. BAAR

**2396. The spectrophotometric determination of
protein at 210 mμ.** M. P. Tombs, F. Souter and
N. F. Maclagan (Dept. of Chem. Path., Westminster
Med. Sch., London, England). *Biochem. J.*, 1959,
73 (1), 167–171.—The total proteins of normal and
pathological sera have mean specific extinction
coeff. $E_{1cm}^{0.1\%}$ of 20·5 ± 0·14. For the determina-
tion of total serum proteins, the serum (0·1 ml) is
diluted to 200 ml with 0·9% aq. NaCl soln. and
the extinction is measured spectrophotometrically
at 210 mμ within 2 hr. of dilution. Albumin to
globulin ratios are determined similarly after pptn.
of the globulins with $(NH_4)_2SO_4$ or Na_2SO_4. The
method is applicable to all proteins studied except
those that contain unusually large amounts of
aromatic amino acids. The use of the extinction at
210 mμ for the detection of very small amounts of
protein (2 μg per ml) and its application to column
hromatography are discussed. J. N. ASHLEY

**2397. Photo-electric ultra-violet photometry ap-
plied to protein electrophoresis.** N. Ressler (Wayne
County Gen. Hosp., Eloise, Mich., U.S.A.). *J. Lab.
Clin. Med.*, 1959, **54** (2), 291–299.—Electrophoresis
is carried out on a quartz or silica plate as
previously described (*Ibid.*, 1959, **54**, 115). The
migration of the proteins is followed by means of
a photo-electric cell that can be moved horizontally
under the plate, which is illuminated with mono-
chromatic u.v. radiation from above. The results
are compared with those by the photographic
method described previously. W. H. C. SHAW

**2398. Influence of the supporting medium on the
fractionation of proteins by zone electrophoresis.**
E. E. Miller and P. Bernfeld (Bio-Res. Inst., Cam-
bridge, Mass., U.S.A.). *J. Chromatography*, 1959,
2 (5), 519–524 (in English).—The fractionation of
mouse plasma by zone electrophoresis on potato-
starch granules (**I**) and on corn-starch gel (**II**) has
been studied. The α_2-globulins migrate more slowly
than the β-globulin on **II** than on **I**. The α_1-globulin
migrates ahead of albumin at pH 7·0, but is not
separated from it at pH 8·6 on either **I** or **II**. A
combination of **I** with sections of **II** at the electrode
ends yields the **I** type of fractionation, but prevents
electro-osmotic flow, thus retaining the advantage
of **II**. Because of the presence of acidic impurities
in **I**, the granules must be thoroughly washed and
equilibrated with buffer soln. before use.

A. R. ROGERS

**2399. Determination of urinary glycoprotein
soluble in perchloric acid.** E. Bonomo (Ist. di Clin.
Med., Univ. Milano, Italy). *Diagn., Lab. e Clin.*,
1958, **14** (4), 159–164.—The method is based on
denaturation of the protein that is soluble in
$HClO_4$ by treatment with tungstophosphoric acid
and determination of tyrosine and hexoses by the
methods of Winzler (*cf.* "Methods of Biochemical
Analysis," Interscience Publ., New York, 1955, p.
279). *Procedure*—Dialyse urine (25 ml) against tap
water for 72 hr. and against distilled water for 48 hr.
To the dialysed urine (20 ml) add slowly, with
shaking, 3·6 M $HClO_4$ (4 ml). Set aside for 30 min.
and centrifuge. To an aliquot (8 ml) add a 5%
soln. of tungstophosphoric acid in 2 N HCl (2 ml),
shake and set the mixture aside overnight in the
refrigerator. Centrifuge, and determine the tyro-
sine equivalent and hexose content of the ppt.
Results obtained for normal subjects, and those with
hypermucoprotidaemia and proteinuria were 4·9 ±
1·7, 9·0 ± 3·1, and 19·3 ± 9·5 mg of tyrosine
excreted per 24 hr., and 14·0 ± 5·8, 25·1 ± 7·0, and
47·2 ± 25·2 mg of hexose excreted per 24 hr.,
respectively. E. C. APLING

**2400. Rapid electrophoretic separation of haemo-
globins.** B. Zak, E. M. Eggers, T. L. Jarkowski and
L. A. Williams (Wayne State Univ. Coll. of Med.,
Detroit, Mich., U.S.A.). *J. Lab. Clin. Med.*, 1959,
54 (2), 288–290.—By carrying out the electrophoresis
in highly purified agar gel with a buffer of decreased
ionic strength and a high potential gradient,
separation can be attained in 15 min. The results
agree with those by conventional electrophoresis
and permit the rapid qual. identification of normal
and abnormal haemoglobins. W. H. C. SHAW

**2401. Use of an artificial standard for haemoglobin
determination.** N. Ressler, N. A. Nelson and I. M.
Smith (Wayne County Gen. Hosp., Eloise, Mich.,
U.S.A.). *J. Lab. Clin. Med.*, 1959, **54** (2), 304–310.

—The proposed standard for use in the cyan-methaemoglobin (I) method is a stable soln. containing $NiCl_2.6H_2O$ (43·3 g) and $CoCl_2.6H_2O$ (10·3 g) dissolved in 100 ml of 10% acetic acid. The extinction is determined at 542 mμ in a spectrophotometer having a half-intensity band-width of $\geqslant$ 5 mμ, and is equated with a concn. of 60·1 mg of I per 100 ml. Results on samples are then calculated by application of Beer's law. Over the range 540 to 545 mμ the absorption curve of the standard is almost identical with that of I.

W. H. C. Shaw

2402. Separation of different types of human haemoglobin. H. K. Prins (Central Lab. of the Netherlands Red Cross Blood Transfusion Service, Amsterdam). *J. Chromatography*, 1959, **2** (5), 445–486 (in English).—Electrophoresis, column chromatography, solubility and alkaline denaturation methods for the identification and isolation of normal and abnormal human haemoglobins are reviewed, with 84 references.   A. R. Rogers

2403. Paper-electrophoretic determination of fibrinogen. B. L. Antonaci, L. Del Giovane and G. Macagnino (Ist. di Semeiotica Medica, Univ. Siena, Italy). *Diagn., Lab. e Clin.*, 1958, **14** (10), 429–444.—Serum and plasma from 100 subjects affected by various pathological conditions were examined by paper electrophoresis by the method of Hirsch and Cattaneo (*cf. Arch. Biochem. Biophys.*, 1956, **61**, 27), and the fibrinogen was also determined by measurement of the tyrosine equivalent. Electrophoretic measurements were made by planimetry, subdividing the fractions according to the method of Wallner and Ulke (*cf. Hoppe-Seyl. Z.*, 1952, **290**, 81). Differences between chemical and electrophoretic measurements were from 5 to 80 mg per 100 ml in 80% of cases, and over 130 mg per 100 ml in 8 cases. Statistical analysis showed that differences were random. The results show the necessity for extreme care in the electrophoretic determination of fibrinogen and the rather low accuracy attainable.   E. C. Apling

2404. Determination of plasma fibrinogen. P. Jannella (Ist. Chim. Biol., Univ., Naples). *Biochim. Appl.*, 1959, **6** (4), 180–186.—Available methods are reviewed and a simple and rapid method, based on fractionation of plasma with a saturated soln. of $(NH_4)_2SO_4$ and application of the biuret colour reaction, is given. (*Cf.* Gornall *et al.*, *J. Biol. Chem.*, 1949, **177**, 751.)   E. C. Apling

2405. Estimation of cholesterol in serum. H. H. Leffler (Professional Lab., Washington, D.C.). *Amer. J. Clin. Path.*, 1959, **31** (4), 310–313.—Serum proteins are pptd., and total cholesterol is simultaneously extracted, by the addition of *iso*-propyl alcohol. Glacial acetic acid and an Fe-H_2SO_4 reagent ($FeCl_3.6H_2O$ 0·1% by wt., H_3PO_4 8% by vol. and H_2SO_4 92% by vol.) are added to an aliquot of the clear extract. A stable colour is produced with an extinction at 540 mμ which is proportional to the amount of cholesterol present. Free cholesterol is pptd. from a second aliquot with digitonin, and the cholesterol is then determined after adding the colour reagent to the washed and dried ppt. The procedure is simple and requires a minimum of time and reagents. Duplicate determinations, in the normal range, agree to within 5 mg per 100 ml of serum.   P. Nicholls

2406. Study of lipids in the cerebrospinal fluid. IV. The determination of free and total cholesterol. W. W. Tourtellottee, B. A. Skrentny and R. N. DeJong (Michigan Univ. Med. Sch., Ann Arbor, U.S.A.). *J. Lab. Clin. Med.*, 1959, **54** (2), 197–206. —The described modification of the acetic anhydride - H_2SO_4 method is suitable for the determination of 1 μg of free cholesterol (I) and 0·5 μg of total cholesterol (II) in <2 ml of normal cerebrospinal fluid. After co-precipitation of proteins and lipids, the lipids are extracted from the ppt. and I is determined fluorimetrically on an aliquot of the extract. A second aliquot is evaporated to dryness, the residue is dissolved in acetone - ethanol (1:1) and treated with digitonin in aq. ethanol. The separated ppt. is treated with acetic acid - H_2SO_4 and the colour due to II is measured at 625 mμ. Coefficients of variation are $\simeq \pm 3$ and ± 7% for I and II, respectively.   W. H. C. Shaw

2407. Techniques for separation of plasma cholesterol esters for determination of iodine value and of cholesterol. G. D. Michaels, G. Fukayama, H. P. Chin and P. Wheeler (Inst. for Metabolic Res., Highland-Alameda County Hosp., Oakland, Calif., U.S.A.). *Proc. Soc. Exp. Biol. Med.*, 1958, **98**, 826–829.—A modification of the technique of Schoenheimer and Sperry (*J. Biol. Chem.*, 1934, **106**, 745) is presented; a colorimetric method instead of the less stable Liebermann - Burchard reaction is used.   B. P. Block

2408. Effect of ester formation on the results of cholesterol determination according to Riffart and Keller. C. H. Brieskorn and H. Herrig (Inst. f. Pharm. u. Lebensmittelchem., Univ., Münster i. Westf., Germany). *Z. LebensmittUntersuch.*, 1959, **110** (1), 15–18.—In this reaction (*Ibid.*, 1934, **68**, 113), the rate and intensity of colour development were greater with cholesterol esters than with the equiv. quantities of cholesterol. The sample soln. (containing 40 mg of cholesterol or its ester per 100 ml of ethyl acetate) (4 ml) was mixed with 2 ml of the reagent [a mixture of acetic anhydride (20 ml) and conc. H_2SO_4 (4 ml)]; 1 ml of ethyl acetate was added, and the mixture was maintained at 20°. After 13 min. the extinction was measured at 625 mμ, followed by other readings at 15- or 20-min. intervals. Extinction curves are given for cholesterol and 5 esters.

S. C. I. Abstr.

2409. Serum cholesterol determinations as affected by vitamin A. L. J. Kinley and R. F. Krause (Dept. of Biochem., W. Virginia Univ. Med. Centre, Moyaxtown, U.S.A.). *Proc. Soc. Exp. Biol. Med.*, 1958, **99**, 244–245.—Total cholesterol determinations were made on serum by using the method of Zak (*Amer. J. Clin. Path.*, 1957, **27**, 589). On the addition of 30 μg of vitamin A per 100 ml of serum there was an increase of 10 to 15 μg of total cholesterol per 100 ml. The addition of 120 to 150 μg gave increases of 100 to 200 μg of total cholesterol per 100 ml. Vitamin A, even in amounts of 150 μg per 100 ml, had no effect on the result if the saponification - extraction process of Schoenheimer and Sperry (*J. Biol. Chem.*, 1934, **106**, 745) was used. Thus high vitamin A levels in the serum will cause interference with cholesterol determinations when the Zak method is used, and care in the choice of method is essential where pretreatment with vitamin A is suspected.   B. P. Block

2410. Application of thin-layer chromatography to steroids. M. Barbier, H. Jäger, H. Tobias and E. Wyss (Univ. Basel, Switzerland). *Helv. Chim. Acta*, 1959, **42** (7), 2440–2446 (in German).—Pyrex-glass plates, 15 cm × 20 cm and 5 mm thick, coated with a layer of "Kieselgel G" (Merck A.G.) 0·3 mm thick, are used to separate weakly polar steroids. The preparation of these chromatoplates is described, and the R_F values obtained with them for 22 steroids are quoted. The developing solvent is acetic acid - *cyclo*hexane (3:7 or 3:17), and the spray reagent may be $SbCl_3$ in $CHCl_3$, molybdo-phosphoric acid in ethanol or, for carbonyl compounds, 2:4-dinitrophenylhydrazine in HCl (1:1).
H. M.

2411. Microscopic identification and polymorphism of sex hormones. M. Brandstätter-Kuhnert and A. Kofler (Univ., Innsbruck, Austria). *Mikrochim. Acta*, 1959, (6), 847–853 (in German).—Characterisation of 16 sex hormones and of synthetic compounds having similar effects was carried out by the determination of the usual Kofler constants. Additional reactions assisting identification are given in some cases.
D. F. PHILLIPS

2412. Rapid separation and quantitation of three phenolic oestrogens by paper chromatography. C. W. Porter, E. L. Clark and G. E. Block (Michigan Univ., Ann Arbor, U.S.A.). *J. Lab. Clin. Med.*, 1959, **54** (3), 471–475.—Separation of oestriol, oestradiol and oestrone is attained by descending chromatography on Whatman No. 3 paper (extracted with methanol) for 20 hr. at 35° to 37·5° with the upper phase of a mixture of n-heptane and methanol (1:1). The developed chromatograms are dipped in a freshly prepared mixture (1:1) of 1% aq. $FeCl_3$ soln. and 1% aq. $K_3Fe(CN)_6$ soln. and, immediately after colour development, are washed first with 10% aq. HCl soln. and then with water. While still wet the blue areas are outlined in pencil and when dried the area of the spots is measured with a planimeter. Calibration curves (10 to 30 μg of sample) are linear and recoveries from urine averaged 45 to 67, 94 and 97%, respectively.
W. H. C. SHAW

2413. Phosphoryl chloride enhancement of fluorescence and absorbance of oestrogens in sulphuric acid. J. C. Touchstone, J. W. Greene, jun., and W. R. Kukovetz (Dept. Obstetrics and Gynaecology, Sch. of Med., Univ. of Pennsylvania, Philadelphia, U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1693–1696.—Various concn. of phosphoryl chloride in 88%(v/v) H_2SO_4 were investigated in a study of the fluorescence spectra of oestrone, 17β-oestradiol and oestriol. The optimum conditions for oestrone and oestradiol were the use of 30% phosphoryl chloride in 88% H_2SO_4 and heating at 65° for 30 min. The resulting soln. can be used directly for fluorimetry. The intensity of absorption and fluorescence is approx. trebled for oestrone and oestradiol and greatly increased for oestriol.
S. BAAR

2414. Detection of enzymes by paper chromatography. A. Panek (Lab. of Microbiol., Sch. of Chem., Rio de Janeiro, Brazil). *J. Chromatography*, 1959, **2** (5), 566–568 (in English).—Paper-chromatographic methods are described for the identification of enzymes produced by micro-organisms. The enzymes are partially purified before application of the following tests—amylase: detect the hydrolytic action of the enzyme on starch; aspartase: identify

aspartic acid formed by action of the enzyme on fumaric acid; glucose oxidase: identify gluconic acid formed by action of the enzyme on glucose; maltase: identify glucose formed by action of the enzyme on maltose; transaminase: identify aspartic acid formed by action of the enzyme on alanine and α-oxoglutaric acid in the presence of pyridoxal phosphate.
A. R. ROGERS

2415. Colour detection of peroxidase on electropherograms. H. F. Linskens and J. A. M. Schrauwen (Botan. Lab., Univ. Nijmegen, Netherlands). *J. Chromatography*, 1959, **2** (5), 578 (in German).—Dissolve benzidine (50 mg) and guaiacol (135 mg) in 10% acetic acid (25 ml) with the aid of heat; to 0·25 ml of this soln. add 0·2 M Na acetate (2 ml), 0·005 M $MnSO_4$ (0·1 ml) and 0·12% H_2O_2 soln. (0·5 ml). Spray the air-dried paper electropherogram with this reagent and set it aside for 5 min. in diffused light; fractions containing peroxidase are revealed as red spots. The procedure may be adapted to quant. analysis.
A. R. ROGERS

3416. Routine method for the estimation of lactic dehydrogenase activity. J. King (North Lonsdale Hosp., Barrow-in-Furness, England). *J. Med. Lab. Technol.*, 1959, **16** (4), 265–272.—A buffered substrate is prepared by mixing 70% Na lactate soln. (10 ml), 0·1 N NaOH (75 ml) and 0·1 M glycine buffer (0·7505% of glycine and 0·585% of NaCl) (125 ml). Buffered substrate (1·0 ml) and serum (0·1 ml of a 1 in 5 aq. dilution) are placed in two test-tubes (test and blank), water (0·2 ml) is added to the blank and the tubes are warmed to 37°. To the test is added 0·5% diphosphopyridine nucleotide soln. (0·2 ml) and, after 15 min., 0·02% 2:4-dinitrophenylhydrazine (**I**) soln. in N HCl (1·0 ml) is added to each tube. After a further 15 min. at 37°, 0·4 N NaOH (10 ml) is added to each tube and the extinctions are read at 440 mμ within 5 min. A standard curve is prepared as follows. To a series of tubes containing 0·05 to 0·55 ml of a 0·011% soln. of Na pyruvate in buffered substrate, made up to 1·0 ml with substrate (=250 to 2750 units per 100 ml) are added water (0·3 ml), 0·02% **I** soln. (1·0 ml), and, after 15 min. at 37°, 0·4 N NaOH (10 ml). The extinctions are read at 440 mμ and the results plotted.
R. A. BRENAN

2417. Portable micro-method for the determination of alkaline phosphatase in blood serum. K. Kuroda, M. Fujino and S. Kato (Dept. Biochem., Sch. of Med., Tokushima Univ., Japan). *Tokushima J. Exp. Med.*, 1958, **5**, 102–110.—Blood serum (50 to 100 mg) was collected by the method of Kuroda (*Med. Biol.*, 1951, **10**, 21) in capillary pipettes, which were then sealed and preserved in a freezing mixture without change of alkaline phosphatase activity. Water (4 ml) was added to $\simeq$100 mg of serum in a tube with a sharp-pointed bottom and the capillary tube was destroyed. The supernatant fluid (1 ml) was incubated at 37° with 0·1 M barbitone buffer (1 ml), 0·0025 M disodium p-nitrophenyl phosphate (1 ml) and water (2 ml); after 1 hr. satd. Na_2CO_3 soln. (5 ml) was added and the extinction was measured at 400 mμ. Alternatively, by a more precise method, 0·1 M bicarbonate buffer (1 ml) was used and the reaction was stopped with 0·02 N NaOH (8 ml). The alkaline phosphatase activity was expressed in millimoles of p-nitrophenol liberated by 1000 g of serum at 37° in 1 hr.
NUTR. ABSTR. REV.

2418. New assay for prostatic acid phosphatase in serum. A. L. Babson and P. A. Read (Dept. of Biochem., Warner-Lambert Res. Inst., Morris Plains, N.J., U.S.A.). *Amer. J. Clin. Path.*, 1959, **32** (1), 88–91.—The method is based on the determination of the 1-naphthol, liberated by the enzymatic hydrolysis of 1-naphthyl phosphate, by coupling with tetrazotised *o*-dianisidine and measuring the resulting azo dye colorimetrically. This simple and rapid procedure is specific for prostatic acid phosphatase.   P. NICHOLLS

2419. Some experiences with the determination of pseudocholinesterase according to Vincent's method. P. N. Meulendijk (St. Elisabeths Gasthuis, Arnhem, Netherlands). *Pharm. Weekbl.*, 1959, **94** (19), 623–626.—The standard deviation of a single determination of the percentage of hydrolysis by Vincent's method (*Clin. Chim. Acta*, 1958, **3**, 104) is $\pm 1\%$ (47 duplicate results). The method is also applicable to the determination of specific cholinesterase activity in erythrocytes. *Procedure for determination of pseudocholinesterase in serum*— Dilute the serum 20 times with barbiturate buffer (pH 7·4). Mix 1 ml of this soln. with 1 ml of buffered substrate [1% aq. acetylcholine hydrochloride soln., diluted (1:10) with buffer] and incubate for 30 min. at 37°. Then add 0·2 ml of *N* HCl and 0·4 ml of hydroxylamine soln. (1 vol. of 14% hydroxyammonium chloride soln. mixed with 1 vol. of 14% NaOH soln.; prepared shortly before use) and after 1 min. add 0·2 ml of 4 *N* HCl, 0·2 ml of 0·37 *M* FeCl₃ and 5 ml of H₂O. Measure the extinction at 540 mμ against a blank prepared with 1 ml of buffer instead of 1 ml of buffered substrate. *Determination of specific cholinesterase activity in erythrocytes*—Wash the cells from 0·02 ml of blood twice with 1 ml of physiological salt soln., centrifuging for 5 min. at 3000 r.p.m. Haemolyse the cells by the addition of 1 drop of 0·01% saponin soln. Add 1 ml of buffer and 1 ml of buffered substrate and proceed as described above. Correct the measured extinction for the extinction of the haemolysate ($\simeq$ 0·2 in a 1-cm cell).   M. J. MAURICE

2420. Determination of transaminases. Problems of methodology. F. Candura (Ist. di Med. del Lavoro, Univ., Pavia, Italy). *Diagn., Lab. e Clin.*, 1959, **15** (1), 21–29.—A method of correcting results obtained by the method of Reitman and Frankel (*Amer. J. Clin. Path.*, 1957, **28**, 56) for the interfering effects of co-existing enzyme systems is described. In the modified method, duplicate series of aliquots of substrate are incubated, with the addition of keto acid at the commencement (test) and after incubation (blank). Comparison of blank and test for each of the specific keto acids involved permits the quant. evaluation of the enzymic reactions taking place, and the correction of the estimated transaminase activity. Graphs are presented illustrating the application of the procedure to the interference of oxoglutaric acid in the determination of glutamic - pyruvic transaminase. A theoretical conversion factor (1 unit of transaminase activity = 48·3 × 10⁻⁵ μmole of keto acid produced in 30 min. at 37°) is derived and confirmed experimentally.   E. C. APLING

2421. Determination of lysozyme. Validity of the method of Fogelson. V. Pipitone and R. Russo (Ist. di Patol. spec. Med., Univ., Bari, Italy). *Diagn., Lab. e Clin.*, 1959, **15** (2), 66–74.—A simple modification of the method of Fogelson (*Amer. J. Dig. Dis.*, 1954, **21**, 327) is described.   E. C. APLING

See also *Abstracts*—**2078**, Calcium and Mg in serum. **2111, 2112, 2113**, Determination of ¹⁴C. **2219**, Fluorine in animal organs. **2259**, Amino-N determination. **2276**, Determination of steroids. **2428**, Alkaline oxidation of adrenaline. **2442**, Determination of P2S in body fluids. **2521**, Analysis of labelled amino acids. **2579**, Oxygen in nutrient media. **2581**, Anaerobic pH measurements of blood. **2586**, Gamma spectrometry for mixtures of isotopes.

Pharmaceutical analysis

2422. Determination of morphine in poppy capsules. G. Pruner (Ist. Sup. Sanità, Roma, Italy). *R.C. Ist. Sup. Sanit.*, 1959, **22** (7), 710–716.—The cerimetric method (*Ibid.*, 1956, **19**, 492) has been modified for application to poppy capsules. *Procedure*—The powdered sample (50 g) is ground in a mortar with H₂O (50 ml) for 15 min., then Al₂O₃ (5 g) is added, the mixture is centrifuged for 6 min., and the supernatant liquid is filtered through acid Al₂O₃ (chromatographic) on a sintered glass filter. The residue in the tube is washed with H₂O (40 ml), and centrifuged and filtered as before. The combined filtrates are concentrated, cooled, centrifuged and filtered, and the filtrate is made up to 25 ml, shaken with Ca(OH)₂ (5 g) and filtered. An aliquot (20 ml) of the filtrate is treated with 4 ml of 10% Pb acetate soln. and, after 5 min, with 4 ml of 3·75% Na₂SO₄ soln. and 12 ml of buffer soln. (4·138 g of Na₂HPO₄.2H₂O, 1·415 g of KH₂PO₄ and 4·285 g of anhyd. Na₂CO₃, made up to 1 litre), and made up to 40 ml. An aliquot (10 ml) is shaken twice with 10 ml of diethyl ether, and the ether layers are discarded; 5 ml of the aq. layer is treated with 15 ml of buffer soln. and filtered, and 5 ml of the filtrate is treated at 25° with 1 ml of 1% K₃Fe(CN)₆ soln., and, after 5 min., 2 ml of HCl (1:1), and then titrated with 0·005 *N* Ce(SO₄)₂, with ferroin as indicator.   L. ZANONI

2423. Determination of the alkaloid content of tinctures of nux vomica and aconite with aluminium oxide (Woelm). M. Kuntze and M. Hädicke (Staatl. Inst. für Arzneimittelprüfung, Jena). *Pharm. Zentralh.*, 1959, **98** (8), 444–446.— Apply the sample (10 g of tincture of nux vomica or 20 g of tincture of aconite) to a chromatographic column of alumina (Woelm grade I) (10 or 12 g) and elute with 90% ethanol (50 ml). For nux vomica alkaloids, evaporate the eluate to a vol. of 10 ml; for aconite alkaloids, evaporate to dryness and dissolve the residue in 90% ethanol (10 ml). Add freshly boiled H₂O (10 ml) and titrate with 0·1 *N* HCl, with methyl red as indicator; when the colour starts to change, add freshly boiled H₂O (50 ml) and complete the titration. The results of assays of tinctures by this method are about 7% higher than those by the classical procedure.   A. R. ROGERS

2424. Spectrophotometric method for the estimation of reserpine. B. C. Bose and R. Vijayvargiya (Dept. of Pharmacol., M.G.M. Medical College, Indore, India). *J. Pharm. Pharmacol.*, 1959, **11** (8), 456–461.—Extract a soln. of reserpine (**I**) (1 mg) in CHCl₃ with 0·1 *N* HCl (4 × 10 ml), shaking for 2 to 3 min. each time. Wash the combined extracts

with $CHCl_3$ (5 ml) and reject the aq. phase. Treat the $CHCl_3$ soln. with 0·05 N H_2SO_4 (10 ml) and warm with constant stirring to remove the $CHCl_3$. Extract the acid soln. with benzene (3 × 10 ml), shaking vigorously for 10 min. each time. Extract the combined benzene phases with 0·05 N H_2SO_4 (5 ml) and add the acid extract to the main acid soln. Extract the combined acid soln. with $CHCl_3$ (6 × 10 ml). Wash the combined $CHCl_3$ extracts with 0·1 N HCl (5 ml) and reject the aq. phase. Evaporate the $CHCl_3$ soln. to dryness to remove traces of benzene. Dissolve the residue in $CHCl_3$ and measure the extinction at 268 mμ. Moderate quantities of rescinnamine, serpentine, ajmaline, yohimbine and methyl reserpate cause no appreciable interference. The method has been applied to the determination of **I** in tablets and in crude root extracts with an error of $\simeq \pm 4\%$.

A. R. ROGERS

2425. Determination of particulate matter in concentrated aerosols. Application to analysis of cigarette smoke. W. B. Wartman, jun., E. C. Cogbill and E. S. Harlow (Dept. of Research and Development, The American Tobacco Co., Richmond, Va., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1705–1709.—A simple and reproducible filter system has been devised and used for the collection of smoke particles in cigarette smoke. A glass-fibre filter is used which is capable of retaining particles of 0·3 μ or greater with an efficiency higher than 99·9%. Procedures are described for the determination of nicotine in the filters by both gravimetric and spectrophotometric techniques.

T. R. ANDREW

2426. Microchemical identification of some alkaloids, barbiturates, sulphonamides and new synthetic drugs. I. T. P. A. van der Wegen (Farm. Lab., Rijksuniv., Utrecht, Netherlands). *Pharm. Weekbl.*, 1959, **94** (20), 656–674.—A number of microchemical reactions, based on the formation of characteristic crystals, are described. New reactions are described for 13 compounds, and 42 illustrations of microscope preparations of the crystals formed by these compounds are given.

M. J. MAURICE

2427. Method for the separation and determination of theophylline, theobromine and caffeine. A. J. Shingler and J. K. Carlton (Div. of Sci., Louisiana State Univ., New Orleans, La., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1679–1680.—The aq. sample soln. (2 ml) is mixed with 3·2 g of silicic acid and applied to a silicic acid column (prep. described). The caffeine is eluted with $CHCl_3$ (H_2O-washed) in 5-ml fractions until the extinction of the eluate is the same at 276·5 mμ and 310 mμ. When the caffeine elution is complete, the theophylline and the theobromine are successively eluted with 5% butanol in $CHCl_3$, and the extinctions are again measured at the same wavelengths. The method is accurate in the 10 to 2500-p.p.m. range.

S. BAAR

2428. Influence of copper and EDTA on the alkaline oxidation of adrenaline. J. G. L. Harthon (Anal. Dept., Res. Lab., AB Bofors, Nobelkrut, Sweden). *J. Pharm. Pharmacol.*, 1959, **11** (9), 553–556.—Adrenaline can be quant. determined in the presence of sulphite by measuring the max. fluorescence at 505 mμ obtained by oxidation in alkaline soln. The presence of Cu in the soln. quenches the fluorescence according to the Stern - Volmer law. EDTA in the absence of Cu has no

influence on the magnitude and the rate of development of fluorescence, but the EDTA - Cu complex strongly catalyses the reaction; an increasing concn. of Cu decreases the fluorescence, but not to the same extent as for EDTA-free soln.

A. R. ROGERS

2429. Rapid photometric method for determination of arbutin in drugs and pharmaceutical preparations. K. H. Müller and E. Hackenberg (Pharm.-Wissenschaftl. Abt. II, Arzneimittelwerk A. Nattermann & Cie, Köln-Braunsfeld). *Arzneimittel-Forsch.*, 1959, **9** (8), 529–532.—The method is based on the reaction of arbutin with 4-aminophenazone in the presence of NH_3 and $K_3Fe(CN)_6$. The orange-coloured compound is extracted with $CHCl_3$ and the extinction of the $CHCl_3$ soln. is measured at 455 mμ.

M. H. SAWISTOWSKA

2430. Approximate determination of anthraquinones without expensive apparatus. XII. Anthraquinones. H. Auterhoff and N. Maiss (Pharm.-chem. Inst., Tech. Hochsch., Braunschweig). *Arch. Pharm., Berlin*, 1959, **292** (8–9); *Mitt. dtsch. pharm. Ges.*, 1959, **64** (8–9), 121–127.—Procedures are described for the extraction of drugs (rhubarb and frangula with glacial acetic acid, aloes and cascara with 25% $FeCl_3$ soln. - acetone, and senna with 25% HCl soln. - glacial acetic acid), purification of the extract by solvent extraction and its final transfer to an alkaline soln. The red colour of the alkaline soln. is then read in a Lange colorimeter, or compared visually with mixtures of 0·25 N $CoCl_2$, 0·167 N $FeCl_3$ and 0·25 N $CuSO_4$. The error is $\simeq 10\%$.

M. H. SAWISTOWSKA

2431. A completely automated system for the chemical determination of streptomycin and penicillin in fermentation media. A. Ferrari, F. M. Russo-Alesi and J. M. Kelly (Technicon Instruments Corp., Chauncey, N.Y., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1710–1717.—The standard methods for streptomycin assay, namely the photometric determination of the alkaline degradation product maltol (Eisenman and Bricker, *Ibid.*, 1949, **21**, 1507) and the determination of penicilloic acid by iodimetry (Alicino, *Ind. Eng. Chem., Anal. Ed.*, 1946, **18**, 619), are adapted to automatic analysis. For the determination of streptomycin, a pump system delivers the samples and 1·6 N NaOH at fixed rates to mixing coils. The mixture is then heated, pumped to a cooling coil and then into a dialysis unit, where the diffused maltol meets a stream of the reagent, $FeCl_3$ in 2·2 N HCl. The non-diffusible substances are discarded. The extinction of the reaction product at 530 mμ is automatically recorded. The samples are spaced in the delivery tubes by the introduction of air bubbles. A similar system is used for the determination of penicillin.

S. BAAR

2432. Recommended method for the determination of the capsaicin content of capsicum and its preparations. Pharmaceutical Society of Great Britain and Society for Analytical Chemistry. Joint Committee on Methods of Assay of Crude Drugs. *Analyst*, 1959, **84**, 603–617.—By specified procedures adapted to the nature of the sample an extract in absolute methanol is obtained. Capsaicin is separated from this extract either by a chromatographic procedure or by a diethyl ether - alkali extraction. It is then determined either by u.v. spectrophotometry or by a colorimetric method based on the coupling reaction between diazobenzenesulphonic acid and capsaicin. A method for

the preparation of pure capsaicin is described. The possibility of using more readily available non-pungent standards for calibration in the diazo process is discussed, but hitherto an ideal substitute has not been encountered.   A. O. Jones

2433. Identification of barbiturates by paper chromatography. R. Hilf, G. A. Lightbourn and F. F. Castano (6th U.S. Army Area Med. Lab., Fort Baker, Calif., U.S.A.). *J. Lab. Clin. Med.*, 1959, **54** (2), 320–324.—Chromatography is carried out by the descending technique on Whatman No. 1 paper with *n*-butanol satd. with 5 N aq. NH$_3$. R_F values for 18 commercial barbiturates are listed. The compounds are located and characterised by fluorescence under u.v. light, and by their reactions with silver acetate, KMnO$_4$ and Lemaire's reagent. For the determination, the silver barbiturate spots are cut out, eluted with 10% aq. HCl and purified by extraction into CHCl$_3$ and re-extraction with 0·45 N NaOH; u.v. extinctions are measured in the alkaline soln.   W. H. C. Shaw

2434. Colorimetric method for the determination of Luminal [phenobarbitone] in small amounts of drug mixtures. N. Stamenova and P. Abrasheva (Higher Med. Inst., Sofia). *Nauch. Trud., VMI, Sofia*, 1958, **5** (2), 133–140.—The method is based on the qual. colour reactions of Parri and Griffon and can be used to determine 0·5 to 1 mg of phenobarbitone (**I**) in drug mixtures (1 to 2 g). The following proportions of reagents are the most suitable. (*i*) A 3% ethanolic soln. of cobalt nitrate (2 drops) and a 1% ethanolic soln. of diethanolamine (3 drops) are added to 95% ethanol (1 ml) containing ≯1 mg of **I**. (*ii*) A 5% soln. of cobalt nitrate in methanol (1 drop) and a soln. of NH$_3$ in aq. methanol (4·5 ml of conc. aq. NH$_3$ diluted with methanol to 20 ml) (3 drops) are added to 1 ml of anhyd. methanol containing ≯1 mg of **I**. Standard comparison soln. are prepared from alkaline Na alizarinsulphonate soln. or from a mixture of ethanolic bromophenol blue soln. and aq. Na alizarinsulphonate soln. The method has been used for the determination of **I** in syrups and in powders and pills.   Abstr. Bulg. Sci. Lit.

2435. Ion-exchange method for the determination of antipyrin [phenazone] and caffeine in the presence of phenacetin. W. Kamp (Farm. Lab., Rijksuniv., Utrecht, Netherlands). *Pharm. Weekbl.*, 1959, **94** (16), 521–525.—*Procedure*—Dissolve the mixture of phenazone (**I**), caffeine (**II**) and phenacetin (**III**) in water and pass the solution through a column of Dowex 50-X2 (100 to 200 mesh) (Fe^{III} form) at 70° at a rate of 20 drops per min. Wash the column with water, combine the percolate and washings and evaporate to dryness. Continue the washing of the column until there is no further increase in weight of the residue. The residue (*A*) consists of **II** and **III**. Wash the column with a soln. (3 N in NH$_3$ and 0·1 M in ammonium acetate) until the red colour of the column has disappeared. Evaporate the eluate to ≃10 ml and determine **I** bromatometrically by the method of Schulek and Kovàcs (*Z. anal. Chem.*, 1941, **121**, 17). To the residue *A* add 20 ml of 0·5 N HCl and boil for 1 hr. under reflux. After cooling, dilute with 80 ml of water and pass the soln. through a column of Dowex 50-X2 (H form) at pH 1. Wash the column with 0·1 N HCl and then with water until the eluate is chloride-free. Then wash with a soln. N in acetic acid and 0·1 M in ammonium acetate, evaporate the eluate to dryness and weigh

the residue, of **II**. Wash the column with water until the eluate is neutral and then elute with N NH$_3$ in 96% ethanol. Determine p-phenetidine in the eluate colorimetrically by the method of Brodie and Axelrod (*J. Pharmacol.*, 1949, **97**, 58).   M. J. Maurice

2436. Identification and determination of drugs of the phenothiazine group by chromatography on paper. G. Nadeau and G. Sobolewski (Lab. de Biochem., Hôpital Saint-Michel Archange, Quebec, Canada). *J. Chromatography*, 1959, **2** (5), 544–546 (in French).—Develop on S. & S. No. 576 paper with the aq. phase of a mixture of 5%(NH$_4$)$_2$SO$_4$ soln. -*isobutyl* alcohol (1:1) by ascending chromatography in the manner described previously (*cf.* Nadeau *et al.*, *Anal. Abstr.*, 1959, **6**, 1887). The separated phenothiazines are revealed by potassium iodo-platinate soln. as intense blue bands. For quant. analysis, elute with citrate - phosphate buffer of pH 4·0 (10 ml), add 2% aq. gum ghatti soln. (0·2 ml) and iodoplatinate reagent (0·1 ml) and measure the extinction at 610 mμ.   A. R. Rogers

2437. New method for the determination of benactyzine. M. Sterescu, N. Keim and H. Aftalion (Inst. Chem. Pharm. Res., Bucureşti, Romania). *Rev. Chim., Bucharest*, 1959, **10** (9), 535–536.—Two methods are proposed, based on the formation of a complex of benactyzine with ammonium reineckate. In one, the pptd. complex is dissolved in acetone, and the soln. is treated with AgNO$_3$ soln., the excess of which is titrated. In the second method, the soln. of the complex is determined photometrically, at 530 mμ.   H. Sher

2438. Studies of anticoagulants. XXXI. The paper chromatography of methylated products of Pelentan [ethyl biscoumacetate] and dicoumarol with diazomethane. E. Knobloch, Z. Ledvinová and I. M. Hais (Res. Inst. Pharm. and Biochem., Prague). *Coll. Czech. Chem. Commun.*, 1959, **24** (8), 2832–2833 (in German).—For the chromatographic separation, 500 μg of the methylated product, dissolved in acetone, was applied to the paper, which was then impregnated by immersion in a 50% ethanolic soln. of formamide. The excess of formamide was removed by blotting between filter-paper sheets and, after 30 min., the separation was carried out by the descending technique with light petroleum - benzene (3:2) as solvent. For detection, exposure to chlorine gas followed by spraying with 2% ethanolic KOH soln. (Fučík and Kořistek, *Chem. Listy*, 1952, **46**, 190) is recommended.   J. Zýka

2439. Determination of some oral antidiabetic drugs. J. B. Dave and J. L. Patel (Alnish Dept., Alembic Chem. Works, Baroda, India). *Indian J. Pharm.*, 1959, **21** (8), 226–229.—Dissolve the sample of carbutamide, tolbutamide or chlorpropamide (about 80 mg in the pure state or in powdered tablets) in pyridine (10 ml) and immediately titrate with 0·08 N Na methoxide in methanol - benzene (3:17), with thymol blue as indicator. Agreement with the nitrite and Kjeldahl methods is satisfactory; the error is < ±1%.   A. R. Rogers

2440. Photometric determination of prednisone in admixture with cortisone. B. Kakáč (Res. Inst. Pharm. and Biochem., Prague). *Českosl. Farm.*, 1959, **8** (8), 427–430.—The influence of H$_2$SO$_4$ and acetic acid and of temp. was studied and a new

method was evolved for the determination of prednisone (**I**) in admixture with cortisone (**II**), which is suitable for following the course of the microbiological transformation of **II** into **I** in nutrient media. Large amounts of hydrocortisone, oestrone, oestradiol, stilboestrol, ergosterol and 7-dehydrocholesterol interfere. *Procedure*— Dilute the sample ($\simeq$ 1 mg of **II**) to 5 ml in a 25-ml separating-funnel, add $CHCl_3$ (10 ml) and anhyd. Na_2SO_4 (0·5 to 1 g) and shake for 2 min. Separate the $CHCl_3$ layer and repeat the shaking of the aq. layer with $CHCl_3$ (10 ml). Combine the $CHCl_3$ layers and wash once with 8 ml of 0·1 N NaOH and twice with 10 ml of H_2O. Dry the $CHCl_3$ layer with a small amount of anhyd. Na_2SO_4, filter into a 25-ml flask, wash the filter several times with $CHCl_3$ and dilute the filtrate to volume. Evaporate 5 ml of the soln. in a test-tube on a water bath, add a freshly prepared mixture of acetic acid and H_2SO_4 (5:1) (6 ml), shaked an heat for exactly 1 hr. on a boiling-water bath. Remove the tube from the bath and after 20 min. measure the extinction in a Hilger spectrophotometer with a blue - green filter 603.   J. ZÝKA

2441. Microchemical reactions of sulpha compounds with copper acetate and amines. H. M. Romijn. *Pharm. Weekbl.*, 1959, **94** (19), 617–623.— Mixtures of metal acetates with some amines were investigated for their ability to form specific crystals with sulpha compounds. The best results were obtained with Cu acetate in combination with methylamine, propylamine, ethylenediamine or propylenediamine. The crystals given by 21 sulpha compounds are described and illustrated by 12 photomicrographs.   M. J. MAURICE

2442. 2-Hydroxyiminomethyl-N-methylpyridinium methanesulphonate (P2S), an antidote to organophosphorus poisoning. Its preparation, estimation and stability. N. H. Creasey and A. L. Green (Chem. Defence Exp. Estab., Porton Down, Wilts., England). *J. Pharm. Pharmacol.*, 1959, **11** (8), 485–490.—To determine P2S (**I**) in tissues, blood, urine or faeces, remove proteins from an aq. extract by pptn. with $Ba(OH)_2$ and $ZnSO_4$, mix a 3-ml aliquot ($\simeq$ 30 µg of **I**) with 20% NaOH soln. (0·2 ml) and measure the extinction at 335 mμ. Apply a correction for the u.v. absorption of the tissue extract. To determine CN^-, a decomposition product, in samples of **I**, separate the CN^- in a Conway cell before determining it by the colorimetric method of Aldridge (*Analyst*, 1945, **70**, 474); alternatively, apply a correction for the colour given by **I** itself.   A. R. ROGERS

2443. Azeotropic distillation as a method of drug control. IV. Determination of ethanol by distillation of the ternary mixture of ethanol, benzene and water. B. Melichar, J. Horka, E. Nečesaná and D. Malá (Dept. Pharm. Chem., Pharm. Fac., Brno, Czechoslovakia). *Českosl. Farm.*, 1959, **8** (8), 423–426.— The apparatus previously described (Melichar and Horka, *Ibid.*, 1954, **3**, 231) is used for the determination of ethanol in the presence of volatile compounds (*e.g.*, menthol and volatile oils). The ternary mixture is distilled off (b.p. 64·86°) and the volume of the distillate is measured (4·96 ml $\equiv$ 1·00 ml of ethanol).   J. ZÝKA

2444. Derivatographic investigation of powders and granulated pharmaceutical preparations. F. Paulik, L. Erdey and G. Takács (Tech. Univ.,

Budapest, Hungary). *Z. anal. Chem.*, 1959, **169** (1), 19–27 (in German).—A procedure is described by which the components of the total water content (bound mechanically, by lattice energy, etc.) of powders and granulated pharmaceutical products can be separated and determined. The apparatus has been described previously (*cf.* Paulik *et al.*, *Z. anal. Chem.*, 1958, **160**, 241), and three quantities are plotted as functions of time, *viz*, temp. of the apparatus, change in weight, and rate of change in weight. The curves derived are plotted as a function of the temp. of the apparatus. Typical curves are shown for several powders and are used to determine the various components of the water content. The decomposition of lactose has also been studied. The method has potential use in the study of other organic decomposition reactions.   S. M. MARSH

See also *Abstracts*—**2258**, Determination of Hg in organic compounds. **2271**, Tartaric acid in presence of citric acid. **2276**, Determination of camphor and steroids. **2287**, Non-aq. sub-micro titration of bases. **2293**, Determination of isoniazid. **2297**, Determination of urethanes and meprobamate. **2316**, Electrophoresis of nicotinamide. **2354**, Phenazone in plasma. **2363**, Cobalt in vitamin preparations. **2365**, Sulphafurazole and sulphadimethoxine in blood. **2366**, Bromosulphonephthalein in serum. **2376**, Detection of flavanones.

Food

Foods and food additives, beverages, edible oils and fats, vitamins.

2445. Determination of sugar in sugar beets. V. Automation in tare-laboratory procedure. W. H. Parker. *Int. Sugar J.*, 1959, **61**, 231–235.—The brei sample is weighed to within 1 g of the standard weight on onion-skin-paper squares on a balance which adjusts the vol. of basic Pb acetate to be delivered from an automatic pipette to correspond to the weight of the sample. The brei is transferred to a capsule and the standard rubber-covered disc applied by hand after addition of the Pb acetate. The capsule is placed on an oscillating tray for the 10-min. extraction period. The final shaking and filtering are done by hand. The soln. is drawn through a filter-stick into a 2-cm cell and the sugar content determined to 0·05% in an E.T.L. - N.P.L. saccharimeter. In this instrument, which is described, the Faraday effect is used to adjust the plane of polarisation over a range of 0·5° with a precision of $\pm 0·0002°$ of arc, corresponding to 0·01° I.S.S. The results are punched into Hollerith cards. A reading takes 30 sec.   G. BURGER

2446. Determination of dextran and starch in cane juices and sugar products. R. I. Nicholson and M. Horsley (Res. Dept., Colonial Sugar Refining Co., Ltd., Sydney, Australia). *J. Agric. Food Chem.*, 1959, **7** (9), 640–643.—The method described for the rapid assay of dextran and starch in the presence of sucrose is based on co-pptg. the starch and dextran by ethanol in the presence of salts, dissolving the ppt. by digestion with formamide, and determining starch colorimetrically with iodine on one aliquot of the soln., and dextran on a second aliquot by measurement of the haze produced by ethanol in the presence of trichloroacetic acid, after removal of starch with salivary amylase at

pH 6·9. Both impurities are of importance in sugar refineries, starch being associated with problems of filtration, and dextran in sugar soln. indicating delay in harvesting or spoilage of juice. Standard curves are constructed with sugar cane or potato starch, and with dextran from *Leuconostoc mesenteroides*.   M. D. ANDERSON

2447. Rapid paper-chromatographic determination of invert sugar in sugar-factory juices. V. Prey and F. Stadler. *Z. Zuckerind.*, 1959, **9**, 286–289.—The method occupies ≃80 min. Calcined glass-fibre paper impregnated with water glass is used, with phenol - water - acetone - ether as solvent. The amount of invert sugar is determined colorimetrically as the formazan by reaction with a triphenyltetrazolium chloride soln. Sucrose does not interfere.   SUGAR IND. ABSTR.

2448. Quantitative determination of non-nitrogenous organic acids in sugar-factory juices. V. Prey, O. Szabolcs and I. Szabolcs. *Z. Zuckerind.*, 1959, **9**, 192–196.—The juices are treated by ion exchangers, and the eluate from the anion exchanger, containing pyrrolidonecarboxylic acid and nonnitrogenous acids, is analysed by one- or two-dimensional paper chromatography, with methanol - aq. NH_3 or, after acidification, with ethyl acetate - acetic acid - water mixtures as solvent. R_F values are tabulated. The spots are measured by a planimeter, and the amounts present can then be calculated.   SUGAR IND. ABSTR.

2449. Detection of added xanthophyll in alimentary pastes. M. Brogioni and U. Franconi (Lab. Chim. Prov. Perugia, Italy). *Chimica, Milano*, 1959, **35** (9), 540–545.—*Procedure*—The milled sample (25 g) in a G3 sintered glass filter fitted with a filter-paper is washed with light petroleum until the filtrate is colourless. The extract is evaporated to 1 ml and the soln. is passed through a chromatographic column (1·2 cm × 15 cm) packed with activated alumina. The column is eluted with light petroleum (4 ml). A pink band below the orange zone (which appears violet under u.v.) indicates the presence of xanthophyll.   L. ZANONI

2450. Determination of the nature of wheat-grain proteins by paper partition chromatography. N. P. Lebedeva. *Nauch. Trudỹ Nauch. Inst. Zemled. Tsentr. Raion. Nechern. Polosỹ.*, 1958, (18), 82–86; *Ref. Zhur., Khim., Biol. Khim.*, 1959, (20), Abstr. No. 26,445.—Descending paper chromatography with butanol - glacial acetic acid - water (4 : 1 : 1 and 8 : 3 : 1) was applied to the amino-acid determination of the proteins of rye gliadin and wheat grain. A flour sample (containing 30 to 50 mg of N) was hydrolysed for 32 hr. at 105° with 6 N HCl in a sealed tube. The acid was removed *in vacuo* and the residue dried over KOH in a desiccator. The hydrolysate was freed from humic compounds by centrifuging. The chromatographic paper was given a preliminary wash in 0·1% 8-hydroxyquinoline soln. The hydrolysate (≃40 to 200 μg of protein) was transferred to the paper and subjected to a 5-fold development with one of the above solvent mixtures; the chromatograms were dried and sprayed in a stream of N with 0·5% butanolic ninhydrin soln. After 18 to 20 hr. the chromatograms were sectioned and the coloured zones eluted with methanol. A 5% ethanolic $Cu(NO_3)_2$ soln. was added dropwise to the eluates and colorimetric measurements were made. It was possible to obtain a sharp separation of 16 amino acids. It

was shown that rye gliadin contains all the essential amino acids. A higher lysine content was obtained than is generally reported.   K. R. C.

2451. Orange-G binding as a measure of protein content. J. Bunyan (Walton Oaks Exper. Station, Vitamins Ltd., Tadworth, Surrey, England). *J. Sci. Food Agric.*, 1959, **10** (8), 425–430.—Some correlation between crude protein content and degree of dye-binding with Orange G (C.I. Acid Orange 10) has been found for meals made from meat, whale meat, fish, soya beans and groundnuts, although a few meat and whale-meat meals were atypical. Reproducibility for any given meal was good, but variations between meals of the same type reduced the value of the test. In all cases, dye-binding was due in part to constituents other than protein.

P. D. PARR-RICHARD

2452. Preservation of milk samples and the analysis of altered milk. I. The preservation of milk samples. P. Navellier. *Ann. Falsif.*, 1959, **52**, 361–368.—A collaborative study of methods of preservation and their effects on the composition and analysis of stored milk samples is reported. The preferred method of preservation is with formaldehyde, combined when possible with storage at low temperature (4° to 5°). It is recommended that preservation with $K_2Cr_2O_7$ should be abandoned.   E. C. APLING

2453. Photometric milk-fat determination. G. Haugaard and J. D. Pettinati (Nat. Dairy Prod. Corp., Oakdale, Long Island, N.Y., U.S.A.). *J. Dairy Sci.*, 1959, **42** (8), 1255–1275.—The method is based on measurement of the parallel and total transmitted light of the prepared sample. After preliminary homogenisation, interference by casein micelles is avoided by the addition of a Ca-chelating agent and coherent scattering by fivefold dilution. Two linear equations relating scattering and the two remaining variables, fat content and mean globule size, are derived. From results on samples of known fat content a nomogram is constructed from which the fat content of samples can be read off directly from the observations. Good agreement with fat determined by the Babcock method is attained on commercial milk samples, and the application of the method to other dairy products is discussed.

W. H. C. SHAW

2454. Butter-fat oxidation. Evaluation of Lea's aldehyde determination method. A. Tamsma and R. D. Powell (Dairy Ind. Sect., Iowa Agric. Exp. Sta., Ames, U.S.A.). *J. Agric. Food Chem.*, 1959, **7** (9), 643–646.—The iodimetric method of Lea (*Ind. Eng. Chem., Anal. Ed.*, 1934, **6**, 241) for titration of the bisulphite bound by the carbonyl group gave recoveries of 95·4 to 98·9% for *n*-heptaldehyde, but for normal saturated C_6, C_8 and C_{10} aldehydes the recoveries were 94, 63 and 22 to 24%, respectively. Recoveries of C_8 and C_{10} aldehydes were increased to 90 and 74%, respectively, by increasing the time of shaking to 6 hr. Incomplete recoveries may be due to decreased solubility in water and lower reactivity with bisulphite. Unsaturated compounds may give high results owing to reaction of the double bonds. The reaction products and yields were examined by solvent extraction after decomposition of the bisulphite compound. The aldehyde recovered from oxidised milk fat possessed some characters of a saturated ketone; the yield represented a heptaldehyde fraction in the aldehydes of the milk fat of 33%

(Lea's method) or 10% (u.v. absorption at 293 to 294 mμ). Some modifications of Lea's method are described.   M. D. ANDERSON

2455. Analysis of milk phospholipids by chromatography and infra-red spectrophotometry. L. M. Smith and N. K. Freeman (California Univ., Davis, U.S.A.). *J. Dairy Sci.*, 1959, **42** (9), 1450–1462.— Prepared milk phospholipids are fractionated on columns of silicic acid - Celite by successive development with $CHCl_3$, acetone, methanol - $CHCl_3$ mixtures and methanol. Fractions corresponding to cerebrosides, kephalins, lecithins and sphingomyelins are assayed chemically and by i.r. absorption at characteristic wavelengths on soln. in $CHCl_3$ or CS_2. The respective percentages (by wt.) of these lipid classes were 6, 35, 32 and 24, and evidence is presented for the existence of minor constituents. Advantages and limitations of the method are discussed.   W. H. C. SHAW

2456. Nephelo-colorimetric determination of lactose. J. C. Godfrain, P. Bertrand and L. Liandier (Lab. Chim., École Nat. Vet., Toulouse, France). *Lait*, 1959, **39**, 32–36.—To the milk sample (5 ml) were added HCl (1 ml) and 5% $NaPO_3$ soln. (5 ml), and, after being shaken and made up to 100 ml, the mixture was filtered. Water (10 ml) was mixed with the filtrate (0·1 ml), and to a 2-ml aliquot of this soln. were added 0·2% $K_4Fe(CN)_6$ soln. (1 ml) and 1 ml of a soln. containing 1% KCN soln. (300 ml) and 8% Na_2CO_3 soln. (200 ml) per litre. The soln. was heated at 103° to 104° for 25 min. and after rapid cooling 10 ml of a mixture (1:9) of a ferric soln. [$Fe_2(SO_4)_3$ (5 g), 85% H_3PO_4 (75 ml) and water (100 ml)] and 20% Teepol 710 was added. After 5 min. water was added to make the mixture up to 25 ml and the extinction of the resulting Prussian blue was measured and referred to a standard curve obtained with lactose. Values given for human and cow's milk agreed closely with those obtained by the method of Causse-Bonnans.
                                    NUTR. ABSTR. REV.

2457. Direct analysis of lactose in milk and serum. J. R. Marier and M. Boulet (Nat. Res. Council, Ottawa, Canada). *J. Dairy Sci.*, 1959, **42** (8), 1390–1391.—A modification of the phenol - H_2SO_4 colorimetric method is described. *Procedure*—To 2·0 ml of test soln. (10 to 75 μg of lactose), standard soln. or water (blank) add 0·10 ml of aq. phenol soln. (80 g of phenol dissolved at 50° in 20 ml of water). Add 6·0 ml of conc. H_2SO_4 slowly as a layer, then swirl, set aside for 10 min. and measure the colour (stable for 1 hr.) at 490 mμ.
                                    W. H. C. SHAW

2458. Method for the determination of radionuclides in milk ash. G. K. Murthy, L. P. Jarnagin and A. S. Goldin (R. A. Taft San. Engng Center, Cincinnati, Ohio, U.S.A.). *J. Dairy Sci.*, 1959, (**24** 8), 1276–1287.—Methods are given for the chemical separation of total radio-strontium, ^{90}Sr (^{90}Y), ^{140}Ba and ^{137}Cs after the addition of suitable carriers to the ash from 1 litre of milk. Appropriate β- or γ-counting techniques are then applied. Recoveries of 82 ± 2, 77 ± 4, 85 ± 4 and $96 \pm 3\%$, respectively, are attained. The reasons for the losses, and cross interference between the elements, are discussed.   W. H. C. SHAW

2459. Detection of adulteration in black-currant juice. H. Woidich and T. Langer (Forschungsinst. d. Ernährungswirtsch., Wien, Austria). *Fruchtsaft-*

Ind., 1959, **4** (6), 234–238.—A chromatographic method suitable for routine analysis has been developed which distinguishes black-currant juice from other juices and detects adulteration. *Procedure*—Shake the sample (50 ml, adjusted to pH 10 with NaOH) with diethyl ether (3×50 ml) and evaporate the washed and dried extracts on a water bath; dissolve the residue in 0·2 ml of ethanol, transfer a drop (25 μl) of the soln. to a filter-paper and develop by the ascending technique with n-butanol - glacial acetic acid - water (63:27: 10) until the solvent front has risen at least 20 cm. Dry the chromatogram at room temp. and treat it with ammonia fumes. One blue fluorescent spot and two yellow spots (R_F values 0·58 and 0·33) appear under u.v. light. The yellow spot of R_F value 0·33 is characteristic of black-currant juice. The chromatogram of red-currant juice also shows two yellow and one blue spot, but the R_F values of the yellow spots are 0·58 and 0·81. Juices from blackberries, bilberries and raspberries were examined; their chromatograms differed from that of black-currant juice.   I. DICKINSON

2460. Paper-chromatographic determination of small amounts of fatty adulterants of the coconut oil group in chocolate and its preparations. E. Pietschmann (Chem. Untersuchungsanst., Nürnberg, Germany). *Fette, Seif., Anstrichmitt.*, 1959, **61** (8), 682–686.—By conversion of fats into their hydroxamic acid derivatives and treatment of these with $FeCl_3$, small amounts of lauric acid, etc., present in cacao butter can be identified. *Procedure*—Mix 30 mg of sample with 3 ml of M hydroxylamine and heat gently for 45 min. Cool, add 2 ml of methanol and 1 drop of phenolphthalein soln. and acidify with a few drops of glacial acetic acid - tetrahydrofuran (1:4). Run the chromatogram on acetylated paper (35 to 45% of the possible acetyl content) and develop with ethyl acetate - tetrahydrofuran - water (1:8:8). After 2 to 4 hr., dry and spray with $FeCl_3$ soln. [2% in ethanol - n-butanol (1:4)]. Run a comparison chromatogram with cacao butter containing 4% of coconut oil. Other additions to chocolate (*e.g.*, almond oil) do not interfere, but allowance must be made for the lauric acid present in milk chocolate.
                                    P. D. PARR-RICHARD

2461. Determination of foreign fats in cacao products. XI. Critical examination of common methods for the investigation of adulterants of cacao butter and chocolate. A. Purr (Schokoladelab. des Inst. für Lebensmitteltechnol. u. Verpackung, München, Germany). *Fette, Seif., Anstrichmitt.*, 1959, **61** (8), 675–682.—Five methods for detecting foreign fats in cacao butter are reviewed. The fractionation of the fatty material is represented schematically, showing the most suitable method to use for each component. These methods include i.r. spectroscopy, column chromatography, gas chromatography of the methyl esters of the fatty acids obtained by saponification, and the separation of an acetone-soluble glyceride fraction followed by saponification and separation of the fatty acids by paper or column chromatography.
                                    P. D. PARR-RICHARD

2462. Chromatographic separation of dyestuffs used in the food industry. Z. Hájek (České Čokoládovny Orion, Modřany u Prahy, Czechoslovakia). *Průmysl Potravin*, 1959, **10** (9), 496–498. —The best and most reproducible results in the chromatographic separation of dyes used in the

Czechoslovak food industry were obtained by means of an ascending or descending solvent system consisting of 2% of Na citrate in 5% aq. NH_3. All the dyes except indigotin can be separated by this system and the separated spots can be evaluated photometrically. For the separation of indigotin, a solvent system containing butanol is recommended.   J. ZÝKA

2463. Re-examination of a new colorimetric determination of benzoic acid. H. Hadorn and F. S. Doevelaar (Lab. VSK Basel, Switzerland). *Mitt. Lebensmitt. Hyg., Bern,* 1959, **50** (5), 435–445 (in German).—The method of Spanyár *et al.* (*Anal. Abstr.,* 1959, **6**, 1527) is critically examined. In this method, benzoic acid (**I**) is isolated and nitrated and the nitro compound is dissolved in acetone - ethanol. On adding NaOH, a violet colour is formed which is suitable for photometric measurement. Although 3:5-dinitrobenzoic acid is supposed to be formed, a mixture of nitro compounds is generally obtained. The colour reaction is reproducible and obeys the Beer - Lambert law, but the nitration is not reproducible, and various nitro derivatives are formed in parallel experiments. Thus various colour reactions are obtained and the error is therefore $> \pm 10\%$. The method is specific for **I** and thus it is suitable for its identification in doubtful cases, or when only a small amount of impure **I** has been isolated. In other cases the method of Hadorn (*Brit. Abstr. C*, 1951, 420) is more satisfactory (*e.g.,* when > 10 mg of well-crystallised **I** is available).   A. G. PETO

2464. Contribution to the analysis of small quantities of fluorine in foods and water. III. Quantitative fluoride determination. K.-E. Quentin and J. Indinger (Balneolog. Inst. d. Univ., München, Germany). *Z. LebensmittUntersuch.,* 1959, **110** (4), 249–260.—The procedure described and illustrated comprises the pretreatment of the test materials to avoid loss of fluorine (*e.g.,* by ashing in the presence of CaO at 550° to 650°), concentration of the fluoride by steam-distillation, in the presence of perchloric acid, at 135°, and the determination of F by a colorimetric method. Of these, the method with zirconium - alizarin lake and that with zirconium - Eriochrome cyanine R (C.I. Mordant Blue **3**) (*cf.* Megregian, *Anal. Abstr.,* 1954, **1**, 2676) are the most useful in determining F in the range (0 to 60 μg) which may occur in foods or water. The merits of the two processes are discussed. Either may be modified according to the material under test and its F content.   S.C.I. ABSTR.

2465. Application of gas chromatography to the analysis of alcoholic distillates. F. Cacace, M. Ikram and M. L. Stein (Rome Univ., Italy). *Ann. Chim., Roma,* 1959, **49** (9), 1383–1390.—The identification and determination of minor components in alcoholic liquids is carried out by gas chromatography with two columns in series. The first contains didecyl phthalate as stationary phase, and effectively separates *iso*butyl and *iso*amyl alcohols and acetates; the second contains polyoxyethylene glycol, which separates the more volatile components, *e.g.,* ethyl acetate and 1:1-diethoxyethane. Typical results on some samples of brandy are shown.   L. A. O'NEILL

2466. Determination of higher alcohols in beer. P. Kolbach and G. Sack. *Brauerei, Wiss. Beil.,* 1959, **12** (8), 119-126.—After filtration of the sample, the ethanol content is adjusted to 5% and the soln. is distilled. The acetate, aldehyde and ester contents of the distillate are then decomposed by treatment with H_2SO_4 followed by KOH and $AgNO_3$. After boiling gently, the soln. is again distilled, and a portion of the distillate is treated with vanillin - H_2SO_4 reagent at 0°. The extinction of the resulting soln. is measured, with a green filter, and the higher-alcohol content is calculated in terms of *iso*amyl alcohol. Good reproducibility is obtained.   G. P. COOK

2467. Kaffircorn [sorghum] malting and brewing studies. III. Determination of amylases in kaffircorn malts. L. Novellie (Nat. Chem. Res. Lab., S.A. Council for Sci. and Ind. Res., Pretoria). *J. Sci. Food Agric.,* 1959, **10** (8), 441–449.—Modified Amer. Soc. Brew. Chem. methods for the determination of amylases are described. Kaffircorn malts contain less β-amylase than barley malts so that larger samples and a higher extraction temp. (30°) were used. Soluble amylase was determined by diastasis or dextrinisation with an aq. extract of the malt; insoluble amylase (high in some varieties of kaffircorn) was either determined directly on the ground malt or was first solubilised with 2% peptone soln. Reproducibility of all three methods was good, but the values for the peptone diastatic power gave the best measure of potential sugar production during mashing. A new unit of diastatic power is proposed.

P. D. PARR-RICHARD

2468. Determination of δ-resin in hops. Y. Kuroiwa and E. Kokubo. *Rep. Res. Lab. Kirin Brewery Co., Yokohama,* 1958, (1), 13–18.—The water-sol. δ-resin of hops (Walker and Zakomorny, *J. Inst. Brewing,* 1952, **58**, 439) is important since it is produced by the decomposition of humulone during storage, and it contributes to the bitterness of beer. Its gravimetric determination by the method of Abson *et al.* (*J. Inst. Brewing,* 1954, **60**, 42) is critically examined, and a modified gravimetric and u.v. spectrophotometric method are presented. The sample of hops (5 g) is extracted with dry diethyl ether and the extract is filtered through paper layered with anhyd. Na_2SO_4. An aliquot (20 ml) of the filtrate is evaporated to dryness, and the residue is emulsified with water satd. with ether (50 ml). The ether is removed, the liquid is shaken with hexane (3×50 ml), and the aq. layer is filtered to remove insol. resins; an aliquot (20 ml) of the filtrate is evaporated to dryness and the residue is weighed. The δ-resin (%) is given by $12 \cdot 5 \ A/(100 - H)$, where A is the wt. (mg) of the residue and H is the moisture (%) of the hops. For the spectrophotometric method, an aliquot (10 ml) of the ether extract of the hops is treated as before, except that the extraction is with 3×20 ml of hexane. After filtration to remove insol. resin, the filtrate and washings are made up to 250 ml with water, and the extinction of the soln. is measured at 260 mμ and calculated to the extinction (D_0) in terms of dry hops. The δ-resin (%) is given by $[1250 \ D_0/(211 \cdot 5 + 97 \cdot 6 \ D_0)] \times 100/(100-H)$. The development of the method and the derivation of the equations are both discussed. The spectrophotometric method gives slightly higher results than the gravimetric one, but it is rapid and suitable for routine analysis. It is shown that when hops are stored at 5° to 6° and at 30°, in air and in nitrogen, the δ-resin increases in all cases, but the increase is less under anaerobic conditions and at lower temp.

J. INST. BREW. ABSTR.

2469. **Analytical chemistry of wines. VII. Determination of vanadium in wine with 8-hydroxyquinoline.** H. Eschnauer (Anorg.-Chem. Inst. d. Johannes Gutenberg-Univ., Mainz a. Rhein, Germany). *Z. LebensmittUntersuch.*, 1959, **110** (2), 121–126.—The wine (50 ml) is ashed and the ash is fused with Na_2CO_3 and K_2CO_3 and dissolved in water. From the filtered soln. at pH 4·0 (phthalate buffer), V and Fe are extracted by shaking with 0·5% 8-hydroxyquinoline (**I**) soln. in $CHCl_3$. Water is added, the pH is raised to 9·4 (NH_4NO_3 buffer) and after shaking the V is separated quant. in the aq. layer. After washing the aq. layer with $CHCl_3$ and neutralising with HNO_3 (methyl orange paper), the V is re-extracted with 0·1% **I** soln. in $CHCl_3$ and the extinction of this soln. is measured against a 0·1% **I** soln. in $CHCl_3$ at 560 mμ. Of 10 wines of various years, four musts and four young wines of 1958, the musts contained more V than the wines. The V content of wines varied between 0·06 and 0·26 mg of V per litre. S.C.I. ABSTR.

2470. **Analytical chemistry of wines. VIII. Determination of cobalt in wines with 2-nitroso-1-naphthol.** H. Eschnauer (Anorg.-Chem. Inst. d. Johannes Gutenberg-Univ., Mainz a. Rhein, Germany). *Z. LebensmittUntersuch.*, 1959, **110** (3), 196–200.—To the soln. in N HCl of the ash of 500 ml of wine, Na acetate (2 g) is added and dissolved. The soln. should then be at pH $\simeq 7$. To complex Fe, 50% Na citrate soln. is added dropwise until no red colour is given with KSCN. Glacial acetic acid (1 ml), 2-nitroso-1-naphthol (1% in glacial acetic acid) (1 ml) and toluene (8 ml) are added and the mixture is shaken. The toluene layer is shaken with $2 N$ NaOH (2×20 ml), separated and washed with water; the combined NaOH layers and washings are extracted with toluene (3 ml) and the extract is added to the main toluene layer. The extinction is measured at 360 mμ against pure toluene. In 12 wines and grape-juices the Co content varied from 0·5 to 12 μg of Co per litre. S.C.I. ABSTR.

2471. **Determination of formic acid in wine.** W. Diemair and C. Gundermann (Univ.-Inst. f. Lebensmittelchem., Frankfurt a.M., Germany). *Z. LebensmittUntersuch.*, 1959, **110** (4), 261–265.—The official method for the determination of small amounts of formic acid (**I**) in wine is shown to be unsatisfactory. The **I** can be separated by steam-distillation according to the procedure of Fincke (*Ibid.*, 1911, **21**, 1; 1911, **22**, 88; 1912, **23**, 225; 1912, **25**, 387) or by distillation *in vacuo*. An aliquot (2 ml) of the distillate is reduced (ice-cold) with magnesium ribbon (80 mg) and conc. HCl (0·5 ml, added in 0·05-ml portions at intervals of 1 min.). Ice-cold reagent (4 ml of 5% aq. chromotropic acid soln. made up to 100 ml with 15 M H_2SO_4) (5 ml) is slowly added, the mixture is heated for 30 min. in a boiling-water bath, then cooled, and made up to 10 ml with 9 M H_2SO_4, and the extinction is read against water, with a yellow filter S57. Sugars yield **I** in Fincke's procedure, and a constant amount of 0·06 mg is therefore deducted from the result. In the vacuum-distillation, all the **I** is not recovered, and the result is therefore increased by 10%. The sensitivity is 0·14 μg, and the accuracy is $\pm 5\%$ in dessert wines and $\pm 10\%$ in dry wines. S.C.I. ABSTR.

2472. **Foreign substances in foodstuffs. I. The determination of copper in spirits using Wofatit F.** A. Popov. *Compt. Rend. Acad. Bulg. Sci.*, 1959, **12** (3), 223–226 (in Russian).—A rapid method has

been developed for the determination of Cu in spirits (*e.g.*, vodka, brandy), whether freshly distilled (colourless) or artificially coloured. *Procedure*—Pass the alcoholic drink, containing approx. 15 mg of Cu per litre, through an ion-exchange column (5 cm $\times$ 1 cm) of Wofatit F at a flow rate of 100 ml per hr. and wash the column with 40% ethanol (10 to 20 ml). Elute the copper with 10% HCl (10 ml), then 1% HCl (30 ml) and finally H_2O (20 ml). Make up the eluate to 100 ml, and transfer 3 ml (containing 5 to 60 mg of Cu per litre) to a 50-ml flask, add 50% ammonium acetate buffer soln. (10 ml), make up to 49 ml with water, add 0·1% ethanolic dithio-oxamide soln. (0·5 ml) and make up to volume with water. Determine the Cu spectrophotometrically, with a red filter. The accuracy of the method is within $\pm 3\%$.
A. BURWOOD-SMITH

2473. **Spectrophotometry in the fat industry.** J. P. Wolff (École Sup. des Corps Gras, Paris). *Olii Min.*, 1959, **36** (8), 303–306.—Ultra-violet spectrophotometry may be used to give an indication of the degree of oxidation of an oil from the absorption bands at 232 and 270 mμ, which are due to conjugated dienes and trienes arising from the decomposition of hydroperoxides. The effect of bleaching and refining treatments on the absorption is discussed. L. A. O'NEILL

2474. **Ultra-violet spectrophotometry for the recognition of olive oil.** V. Morani and C. Marignoli Colloca (Staz. Chim. Agrar. Sper., Roma). *Olii Min.*, 1959, **36** (8), 327–328.—Virgin olive oil may be clearly differentiated from rectified oils A or B from the specific extinction coefficients (K) at 262, 268 and 274 mμ. When the values are expressed in terms of $\triangle K = 1000[K_{268} - 0.5 (K_{262} + K_{274})]$, it is found that pure olive oils have a $\triangle K$ of <5, rectified A oils of $\simeq 50$, and rectified B oils of >100. L. A. O'NEILL

2475. **Applications of spectrophotometry in the analysis of olive oil. II. Possible contribution of ultra-violet spectrophotometry to the classification of the oils.** A. Montefredine and L. Laporta (Lab. Chim. Provinciale, Pescara, Italy). *Olii Min.*, 1959, **36** (8), 325–326.—The classification of olive oils in terms of the specific extinction coefficients (K) at 232 and 270 mμ is suggested. Typical figures for K_{232} and K_{270} for virgin, rectified A and rectified B oils are, respectively, 3·0 and 0·2; 4·0 and 0·6 to 0·8; and >4 and 2·0. L. A. O'NEILL

2476. **Use of ultra-violet spectrophotometry for the evaluation and identification of olive oil.** A. Uzzan (Inst. Corps Gras, Paris). *Olii Min.*, 1959, **36** (8), 307–311.—The characterisation of olive oil by means of the specific extinction coefficients at 232 and 270 mμ (K_{232} and K_{270}) is discussed. Results for oils of various origins are given in terms of the values of K_{270} and the ratio R ($= K_{232}/K_{270}$). It is proposed that oils be classified into "best" and "good" qualities according as the values of K_{270}, R and the acidity (%) are, respectively, < 0.16, >10 and <1, and <0.2, >9 and <2.0. L. A. O'NEILL

2477. **Micro-determination of the iodine value.** P. Smits (Unilever Res. Lab., Vlaardingen, The Netherlands). *Rec. Trav. Chim. Pays-Bas*, 1959, **78** (9–10), 713–723 (in English).—The method is suitable for fractions of 10 to 100 μg obtained by gas-liquid chromatography. Iodine soln. (2% in

glacial acetic acid) is mixed before use with an equal vol. of 2% Hg acetate in the same solvent, then diluted with acetic acid so that 0·1 ml contains 2 to 2·5 times as much iodine as will be required for addition. After reaction of the sample in the dark with 0·1 ml of reagent for 15 to 60 min., the soln. is diluted to vol. with N-saturated KI soln. (0·05 M in 75% methanol) and the extinction at 375 mμ is compared with that of a similar soln. which contains no sample. When applied to model substances, the coeff. of variation for 4 to 8 determinations are $\simeq 1\%$ (50 to 120 μg) and $\simeq 2\cdot5$ to 4·5% (< 50 μg).

P. D. PARR-RICHARD

2478. Possibility of identifying additions of rectified oils to expressed olive oils. A. Fabris and M. Vitagliano (Ist. Ind. Agrar., Milan Univ.). *Olii Min.*, 1959, **36** (8), 313–324.—Expressed olive oils, of which 469 samples have been examined, are practically free from conjugated triene compounds, although in a few cases small amounts, probably arising from oxidation of the oil, have been found. Rectified oils A and B, on the other hand, contain significant amounts of conjugated trienes, which appear to be formed mainly during the treatment with bleaching earth; additions of 5 or 10% of the rectified oils to virgin olive oils may readily be detected spectroscopically. Conditions leading to the formation of conjugated triene compounds in the oils have been studied.   L. A. O'NEILL

2479. Paper chromatography in the field of fats and oils. XXXI. The separation of mixtures of synthetic and natural triglycerides by paper chromatography. H. P. Kaufmann and Z. Makus (Dtsch. Inst. f. Fettforsch., Münster, Westfalia). *Fette, Seif., Anstrichmitt.*, 1959, **61** (8), 631–636.— The separation of mixtures of synthetic and natural triglycerides is possible on paper impregnated with undecane or paraffin wax with *iso*propyl alcohol - acetic acid - water (20:5:2) or glacial acetic acid as the mobile phase. The separation is strongly influenced by the degree of impregnation. Natural fats were found to contain pairs of components with the same R_F values, and each fat gave a characteristic number of spots, which could be used as a test of purity. Different methods of identification are discussed and a table is given of the colour reactions of a number of dyes with glycerides.

P. D. PARR-RICHARD

2480. Paper chromatography in the analysis of fats. XXXIV. The detection of fatty aldehydes. 5. Qualitative and quantitative paper-chromatographic analysis of fatty aldehydes. H. P. Kaufmann and H. Kirschnek (Dtsch. Inst. für Fettforschung, Münster, Germany). *Fette, Seif., Anstrichmitt.*, 1959, **61** (9), 750–759.—Saturated and unsaturated aldehydes containing 8 to 18 carbon atoms are converted into the 2:4-dinitrophenylhydrazones and separated on paper impregnated with petroleum spirit (boiling-range 150° to 180°), undecane or tetralin. The mobile phases are mixtures of pyridine, formamide, dimethylformamide, nitromethane and methyl cyanide, and development is carried out for 8 to 12 hr. The positions of the separated substances are found by making a photographic print of the paper. R_F values are given for the aldehydes and also for the mercuric acetate adducts of the unsaturated aldehydes. After detection, the separated substances are extracted from the paper with a solvent and determined by polarography, with a supporting electrolyte of 0·1 M LiCl in 82% aq. dioxan. The $E_{\frac{1}{2}}$

for the reduction of the aromatic nitro group is $-0\cdot43$ V *vs.* the S.C.E. The procedure has a precision of $\pm 5\%$ at the 5-μg level.

W. T. CARTER

2481. Paper chromatography in the analysis of fats. XXXV. The detection of fatty impurities in olive oil by means of paper chromatography. H. P. Kaufmann and M. Aparicio (Dtsch. Inst. für Fettforschung, Münster, Germany). *Fette, Seif., Anstrichmitt.*, 1959, **61** (9), 768–770.—The glycerides in the oil are separated by the technique of Kaufmann and Makus (*Anal. Abstr.*, 1960, **7**, 2479). Any adulteration by vegetable oils causes an increase in the number of glycerides on the chromatogram. By this method it is possible to detect 5 to 10% of soya-bean, cottonseed, arachis, sunflower, sesame, rape, corn, linseed, coconut or palm-kernel oil.   W. T. CARTER

2482. Paper chromatography in the analysis of fats. XXXVI. Paper-chromatographic detection of surface-active substances by the "transparence" method. H. P. Kaufmann and G. Walther (Dtsch. Inst. für Fettforschung, Münster, Germany). *Fette, Seif., Anstrichmitt.*, 1959, **61** (9), 782–784.— Sugar fatty acid monoesters and fatty alcohol polyglycol ethers are separated by partition chromatography on paper impregnated with paraffin wax, with methanol - water - ethyl acetate (6:4:1 or 3:2:1) as mobile phase. The separated substances are detected by spraying the chromatogram with water, which makes the paper transparent in the presence of monoglycerides, partial esters of polyhydric alcohols with fatty acids, fatty alcohol polyglycol ethers and certain hydroxy fatty acids. The sensitivity is 2 to 3 μg. Free fatty acids, neutral fatty acid esters and higher fatty alcohols do not react.   W. T. CARTER

2483. Use of infra-red spectrography in the field of fats and oils. II. Quantitative determination of *trans*-unsaturated fatty acids in the presence of *cis*-isomers and saturated compounds. H. P. Kaufmann, F. Volbert and G. Mankel. *Fette, Seif., Anstrichmitt.*, 1959, **61** (8), 643–651.—Oleic, elaidic, erucic and brassidic acids and their methyl esters and barium salts were specially purified, and mixtures of the *cis*- and *trans*-isomers were examined by i.r. spectrography. With the acids, good results were obtained only when ≮30% of the *trans*-isomer was present; with the esters and barium salts, the mixture should contain ≮3·5% and ≮7%, respectively, of the *trans*-isomer. Tests on natural fats showed that structural effects in certain glycerides cause appreciable positive errors in the determination, so that glycerides should be hydrolysed and the fatty acids converted into methyl esters before i.r. examination.   P. D. PARR-RICHARD

2484. Isolation and determination of partially hydrogenated fatty acids as the mercury adducts. K. Schilling (Dansk Fedtforskninginst., Kopenhagen). *Fette, Seif., Anstrichmitt.*, 1959, **61** (9), 765–768.—The acids are esterified, converted into the mercury adducts by treatment with mercuric acetate in methanol, and separated by countercurrent extraction with light petroleum - benzene - methanol - water - acetic acid (6:4:6:3:3, or 3:2:3:1:1). The separation depends on the degree of unsaturation and the chain length of the acid, and is not affected by the configuration or the positions of the double bonds. The separated fractions are analysed for mercury with dithizone,

and the acids can also be recovered for structural analysis. Examples are given of the separation of oleic, linoleic and linolenic acids, erucic and oleic acids, palmitic and erucic acids, and of the acids in partially hydrogenated linseed and herring oils.

W. T. CARTER

2485. Paper chromatography of the saturated fatty acids. M. A. Buchanan (Inst. of Paper Chemistry, Appleton, Wis., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1616–1618.—Apply a $CHCl_3$ soln. of the sample (containing about 2 mg of acids) to a strip of paper impregnated with heavy mineral oil, heat at 80° to 90° for 5 min. and develop with a mixture of glacial acetic acid - 88% aq. formic acid - 30% aq. H_2O_2 (6:1:1) at 37° by the descending technique. Immerse the air-dried chromatogram for 15 min. in a 0·1% soln. of mercuric acetate in 0·05% acetic acid, wash in running water for 45 min., dry at room temp., and spray with a 0·2% soln. of *sym.*-diphenylcarbazide in 95% ethanol. The acids are revealed as purple spots. Small amounts of the even-numbered straight-chain saturated acids from C_{12} to C_{24} are readily separated from each other and from large amounts of unsaturated acids which react with the solvent system to form products of high R_F values. In an alternative procedure, an acetic acid soln. of the sample is treated with a freshly prepared mixture (1:1) of 88% formic acid and 30% H_2O_2, set aside overnight at room temp., diluted with H_2O, and extracted with $CHCl_3$ before chromatographic development with acetic acid - H_2O (17:3).

A. R. ROGERS

2486. Determination of the degree of unsaturation of long-chain fatty acids by gas - liquid chromatography. A. T. James (Nat. Inst. for Med. Res., Mill Hill, London). *J. Chromatography*, 1959, **2** (5), 552–561 (in English).—The number of double bonds and carbon atoms in an unknown fatty acid can be determined by comparison of the log(relative retention volume) on a polar and a non-polar stationary phase in a gas - liquid chromatogram with values established with known acids.

A. R. ROGERS

2487. Gas - liquid chromatography: retention volumes of the methyl esters of fatty acids, with special reference to normal odd-numbered, *iso* and (+)-*anteiso* acids. J. C. Hawke, R. P. Hansen and F. B. Shorland (Fats Res. Lab., D.S.I.R., Wellington, New Zealand). *J. Chromatography*, 1959, **2** (5), 547–551 (in English).—Relative retention volumes of the methyl esters of fatty acids have been determined at 200° and 220° with silicone grease, Apiezon M and polydiethylene glycol succinate as stationary phases. Among the acids studied were normal odd- and even-numbered saturated acids from C_8 to C_{26}, unsaturated normal acids in the range C_{18} to C_{22}, and *iso* and (+)-*anteiso* acids from C_{12} to C_{18}, inclusive.

A. R. ROGERS

2488. Microbiological determination of vitamins of the B group. J. Adrian (Lab. de Biochim. de la Nutr. du C.N.R.S., Bellevue, S.-et-O., France). *Cah. Tech. Centre Nat. Nutr. Aliment.*, 1959, No. 4, 183 pp.—The general techniques are discussed with reference to the organisms used, the extraction of the vitamins, culture methods, the calculation of the results and the specificity of the methods. Published methods for the various vitamins are reviewed, and recommended procedures for the preparation of media for the individual vitamins are detailed in 21 appendices. (523 references.)

H. S. R.

2489. Infra-red absorption spectra for standardising substances used in vitamin-E synthesis. B. G. Savinov. *Fiz. Sb. L'vov. Univ.*, 1957, [3 (8)], 265–267; *Ref. Zhur., Khim., Biol. Khim.*, 1959, (22), Abstr. No. 29,247.—As the yield of synthetic vitamin E [DL-α-tocopherol] (**I**), obtained by the condensation of phytol (**II**) and trimethylquinol, and subsequent purification, depends so much on the purity of the reagents used, the i.r. absorption spectrum method is used to standardise the purity of **II**. An i.r. spectrograph with a Silit resistor incandescing at 1000° is used in conjunction with a rock-salt prism to study the 3 to 15-μ spectrographic region. A 3330 cm^{-1} absorption band of **II** corresponds to the vibration of an ethoxyl group, a deeper 2890 cm^{-1} band corresponds to the vibration frequency of methyl groups, a small 1665 cm^{-1} band corresponds to a single $C=C$ bond; an intense 1449 cm^{-1} band corresponds to the vibration of a C–H bond and a 1362 cm^{-1} band is explained by deformation of methyl group vibrations. The purity of **II** is also checked by the thermo-stability of **I** and its esters. It is shown that after a 10-hr. heating, **I** retains only one band (1449 cm^{-1} max.). Acetic, succinic and nitrobenzoic acid esters of **I** retain unchanged absorption spectra in the i.r. region after a 10-hr. heating.

K. R. C.

See also Abstracts—**2046**, Tartaric acid in wine. **2273**, Refractive indices of pure sucrose soln. **2281**, Determination of citric acid.

Sanitation

Analysis of air, water, sewage, industrial wastes, industrial poisons.

2490. Spectral determination of water vapour in the upper atmosphere. M. S. Kiseleva, B. S. Neporent and V. A. Fursenkov. *Optics and Spectroscopy*, 1959, **6** (6), 522–524; English translation of *Optika i Spektroskopiya*, 1959, **6** (6), 801.—The water content of the atmosphere at various altitudes up to 11 km was determined with a special grating spectrophotometer to measure absorption characteristics at 1·4, 1·9 and 2·6 μ during balloon flights. Data obtained by successive scanning of the appropriate wavelength regions are transmitted back to earth during the free flight of the balloon. The instrument is calibrated in the laboratory, with multi-path cells giving optical paths up to 100 metres long, and water vapour pressures from 0·9 to 10 torr.

B. S. COOPER

2491. Determination of benzene in the presence of its homologues in the atmosphere. R. Vlasák (Regional Hyg. Epid. Dept., Pardubice, Czechoslovakia). *Pracovní Lékařství*, 1959, **11** (8), 418–422.—Two methods were used, *viz.*, the polarographic method based on nitration (*cf.* Šedivec, *Anal. Abstr.*, 1957, **4**, 3021) and a new sensitive colorimetric method based on the reaction of *m*-dinitrobenzene (**I**) with ethyl methyl ketone (**II**) in pyridine. *Procedure for the colorimetric method*—The material from the air sample is absorbed in 2 ml of nitration mixture (*loc. cit.*); an aliquot (1 ml) is transferred to a 100-ml flask and diluted with H_2O (0·5 ml) and the nitro derivatives (except **I**) are oxidised with chromic acid (*loc. cit.*). Dilute the reaction mixture with H_2O (5 ml), cool, add pyridine (10 ml) and 40% NaOH soln. (7·5 ml), transfer to a separating-funnel and extract the **I** into the pyridine layer.

Separate the aq. layer, wash the pyridine layer with 20% NaOH soln. (5 ml), transfer it to a 25-ml flask and add **II** (5 ml); after 10 min. dilute the violet soln. to vol. with pyridine and mix; after 20 min. measure the extinction (S57 filter). Compare with a calibration curve (0 to 100 μg of benzene). An accuracy of $\pm 5\%$ in both methods was achieved. The adsorption of benzene on activated silica gel was also examined and it was found that adsorption and desorption in a stream of nitrogen at 100° to 110°, are both quantitative.

J. ZÝKA

2492. Suggested reduction methods for the determination of nitrates. J. M. Pappenhagen and J. J. Looker (Kenyon Coll., Gambier, Ohio). *J. Amer. Wat. Wks Ass.*, 1959, **51** (8), 1039–1045.—It is shown that NO_3^- reduced by a zinc-copper couple or alkaline $FeSO_4 - AgSO_4$ can be determined by Nessler reagent. The procedures are given. The latter method is quicker and more sensitive, and achieves the quant. reduction of NO_3^- and the recovery of all the NH_3 produced. Both methods are better than the pyridine-pyrazolone method. Ammonia and NO_2^- interfere in all three methods but they can be removed or determined rapidly. Reduction by aluminium foil is unsatisfactory.

O. M. WHITTON

2493. Chlorine determinations in waste waters. I. Evaluation of the cyanogen chloride method. S. Katz and H. Heukelekian (Dept. of Sanitation, Rutgers Univ., N. Brunswick, N.J., U.S.A.). *Sewage Ind. Wastes*, 1959, **31** (9), 1022–1033.—The method, which is applicable over the range from 0·05 to 1·09 mg per litre, involves treatment with KCN or NaCN soln. to produce CNCl, which is then treated with a pyridine-benzidine mixture to give a yellow-orange colour, which is extracted with *n*-butanol and measured at 480 mμ; interferences are few, and those by CN^-, SCN^- and amino acids can be compensated for by a baseline technique or a blank. The results differ widely from those by the amperometric method, but the superiority of either method has not been demonstrated, and there is evidence that they do not measure either the same type of chlorine residual or a constant proportion thereof. The *o*-tolidine and starch-iodide methods are not applicable to industrial wastes, but with tap-water all four methods give comparable results.

O. M. WHITTON

2494. Suggested revision of phenol red method for bromide. E. Goldman and D. Byles (San. Engng Div., Dept. of Water and Power, Los Angeles, Calif.). *J. Amer. Wat. Wks Ass.*, 1959, **51** (8), 1051–1053.—It is shown that, in the phenol red-chloramine T method for bromide, fading of the colour can be eliminated by the addition of 0·5 to 5·0 millimoles of $Na_2S_2O_3$ to the treated sample (5·0 ml); the optimum time between the addition of the chloramine T and the addition of $Na_2S_2O_3$ is 20 $\pm$ 5 min. at 20° to 25°; the best concn. of the chloramine T soln. is 5·0 mg per ml, and the best wavelength for spectrophotometric examination is 590 mμ. The revised method is given.

O. M. WHITTON

2495. Chemical analysis with an ultra-violet filter photometer. IV. Determination of minute amounts of sulphide ions in water. Takeshi Kato and Koichiro Shinra (North College, Osaka Univ., Kita-ku). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (5), 476–477.—Sulphide ions (0·1 to 2 p.p.m.) in water react with the Cu complex of 1-nitroso-2-naphthol (**I**) at pH 5·2 to 6·4 to give free **I**, which is determined photometrically at 366 mμ. The heterogeneous reaction is complete within 3 min. and the working curve is linear for 0·1 to 2 p.p.m. of S^{2-}. There is no interference from Ca, SO_4^{2-}, NO_3^-, PO_4^{3-} (<500 p.p.m. each), Mg (<300), ClO_3^- or Cl^-. Metal ions that form insol. sulphides vitiate the determination. This method is applicable to the analysis of natural water, and to industrial waste water.

K. SAITO

2496. Suggested method for boron determination with 1:1'-dianthrimide. F. H. Rainwater (U.S. Geol. Survey, Washington, D.C.). *J. Amer. Wat. Wks Ass.*, 1959, **51** (8), 1046–1050.—The method of Ellis *et al.* (*Anal. Chem.*, 1949, **21**, 1345), which depends on the production of a blue colour with 1:1'-dianthrimide (**I**) in conc. H_2SO_4, is modified for application to water samples. The sample (5 ml containing <0·005 mg of B) is mixed with 1 ml of conc. H_2SO_4 and evaporated to 1 ml at 90° (overnight); 5 ml of reagent (0·02% of **I** in conc. H_2SO_4) is added, and the mixture is maintained at 90° for 3 hr. It is then cooled and mixed with 10 ml of conc. H_2SO_4, and the extinction is measured against a blank at 620 mμ. The calibration curve is rectilinear in the range 0 to 5 μg of B. Results on standard soln. containing up to 1·00 mg per litre of B gave a standard deviation (18 results) of $\pm$ 0·007 mg per litre. Results for the recovery of 0·2 mg of B per litre, added to 10 natural waters originally containing from 0·2 to 1·05 mg per litre, indicated a precision of $\pm 0·012$ mg per litre.

O. M. WHITTON

2497. Chlorinated insecticides in surface waters. A. A. Rosen and F. M. Middleton (Robert A. Taft Engng Center, Dept. of Health, Educ. and Welfare, Public Health Serv., Cincinnati, Ohio, U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1729–1732.—Small amounts of chlorinated insecticides are concentrated by adsorption on an activated charcoal column (18 in. × 3 in.), which is suitable for use with 5000 gall. of water, and recovered by extraction with $CHCl_3$. The concentrate (0·25 to 0·5 g) in 3 ml of $CHCl_3$ is added to an alumina column (80 to 200 mesh) (14 cm × 1·9 cm) and eluted with $CHCl_3$ until 3 column-volumes have been collected. The eluate is concentrated to 1 ml on a steam bath and finally *in vacuo* at 50°. The residue is dissolved in 0·2 ml of CS_2 and the i.r. spectrum is recorded. The method is of particular value for the qualitative detection of insecticides; aldrin, BHC, chlordane, DDD [1:1-dichloro-2:2-di-(*p*-chlorophenyl)ethane], DDT, dieldrin, endrin and methoxychlor were investigated.

S. BAAR

2498. Simultaneous determination of traces of iron and aluminium in sea water. Hiroshi Hashitani and Katsumi Yamamoto (Japan Atomic Energy Res. Inst., Tokai, Ibaragi-ken). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (7), 727–731.—The extraction of Al and Fe^{III} oxinate with $CHCl_3$ (*cf.* Motojima and Hashitani, *Anal. Abstr.*, 1958, **5**, 2632) was applied to sea water, the masking action of F^- being eliminated with Be. The optimum pH in the presence of Be is 5·0 to 5·5. Copper, Co and Ni are removed by washing the $CHCl_3$ layer with KCN soln. The salinity does not affect the results. *Procedure*—Boil the sample (1 litre) with conc. HCl (5 ml) and $BeSO_4$ soln. (1·3 mg of Be per ml) (10 ml) and filter. Treat a 250-ml portion of the

filtrate with 8-hydroxyquinoline (1% in acetic acid) (3 ml), ammonium acetate (2 M, 3 ml) and aq. NH_3 soln. to adjust the pH to 5, dilute to 400 ml and shake with $CHCl_3$ (10 ml) for 1 min. Wash the $CHCl_3$ layer with KCN soln. (0·1 M in 4 M NH_4Cl, pH 8·5) (10 ml), dry with Na_2SO_4 and measure the extinction at 390 and 470 mμ. Calculate the amount of Fe and Al by an empirical formula.
K. SAITO

See also Abstracts—**2032**, Carbon dioxide in air. **2089**, Boron hydrides in air. **2221**, Bromide and I⁻ in water. **2464**, Fluoride in water. **2584**, Dissolved O in water.

Agricultural analysis

Soil, fertilisers, herbicides, pesticides, animal feeding-stuffs.

2499. The determination of sulphate in superphosphate. A. G. C. Morris and S. J. Bozalek (African Explosives and Chemical Industries Ltd., Res. Dept., Northrand, Transvaal, S. Africa). *Anal. Chim. Acta*, 1959, **21** (3), 215–221 (in English). —The effects of $BaCl_2$ and HCl concn. and the soln. volume on the gravimetric determination of sulphate as $BaSO_4$ have been examined by means of a factorial experiment. With the final method, 20 determinations on 10 samples of superphosphate containing 40·1% of sulphate gave a standard deviation of 0·08.
W. T. CARTER

2500. Determination of nitrofurazone in vegetable and animal tissue. J. Brüggemann and K. Bronsch (Inst. f. Physiol. u. Ernährung der Tiere, Univ. München, Germany). *Z. anal. Chem.*, 1959, **169** (2), 88–102 (in German).—Two methods are examined— (*i*) determination of nitrofurazone (**I**) as 5-nitro-2-furfuraldehyde phenylhydrazone (*cf.* Buzard *et al.*, *Anal. Abstr.*, 1957, **4**, 2300) and (*ii*) column-chromatographic separation of **I** (*cf.* Beckman, *Anal. Abstr.*, 1958, **5**, 3933) and measurement of the red colour after addition of 0·04 N KOH to the soln. of **I**. Method (*i*) is preferable and, for pellets of chicken food, complete extraction of **I** is achieved with dimethylformamide (**II**) at 70° or with ethanol at 60° in 30 min. The absorption max. at 440 mμ permits >2 mg of **I** per 100 ml to be determined (limits of error ±6%). Modification of method (*ii*) permits the recovery of 99·8 ± 0·4 and 98·6 ± 0·5% of **I** on chromatography of extracts of feeding-stuffs and of animal organs or faeces, respectively. The alumina column is pre-treated with **II**; the first solvent is **II** and the eluent and solvent for colour development is 60% ethanol (max. content of **II** is 12%). Colorimetry at 460 mμ permits the determination of 0·7 to 20 mg of **I** per 100 ml in feeding-stuffs and of 14 μg of **I** per g of animal tissue (dry).
J. P. STERN

2501. Determining micro amounts of *iso*propyl phenylcarbamate. M. Montgomery and V. H. Freed (Dept. of Agric. Chem., Oregon State Coll., Corvallis, U.S.A.). *J. Agric. Food Chem.*, 1959, **7** (9), 617–618.—The selective herbicide *iso*propyl phenylcarbamate (**I**) is determined by alkaline hydrolysis, and coupling the resulting aniline with N-1-naphthylethylenediamine dihydrochloride, to give a colour measured at 560 mμ. Amounts of 0·1 p.p.m. in plant tissue can be determined. The direct hydrolysis of plant tissue, avoiding extraction procedures, was successfully carried out with strawberries. The average recovery of added **I**

was 97·5% (coeff. of variation ±2·9% from 8 determinations). The application of the method to the determination of *iso*propyl *m*-chlorophenylcarbamate is suggested.
M. D. ANDERSON

2502. Colorimetric determination of 1-naphthyl methylcarbamate in agricultural crops. R. Miskus, H. T. Gordon and D. A. George (Dept. of Entomology and Parasitology, Univ. of California, Berkeley, U.S.A.). *J. Agric. Food Chem.*, 1959, **7** (9), 613–614.—Residues in plant tissues of the insecticide 1-naphthyl methylcarbamate (Sevin) (**I**) are determined by extracting with $CHCl_3$, evaporating the solvent, dissolving the residue in water - methanol (1 : 1), filtering and making alkaline with NaOH, which hydrolyses **I** to yield 1-naphthol. This is determined by reaction with *p*-nitrophenyl-diazonium fluoroborate, and measurement of the colour at 590 mμ. The method is suitable for 5 to 40 μg of **I**; recoveries of added **I** were 72 to 134%.
M. D. ANDERSON

2503. Determination of some synthetic plant-growth regulators. G. Sandri (Bologna Univ., Italy). *Ann. Chim.*, *Roma*, 1959, **49** (9), 1420–1425. —Maleic hydrazide may be determined volumetrically by oxidation with HIO_4 or $KBrO_3$ in the presence of HCl, each mol. of hydrazide reacting with 4 equiv. of halogen. Cinnamic acid may be similarly determined by reaction with HIO_4, and coumarin with $KBrO_3$.
L. A. O'NEILL

2504. Characterisation and determination of traces of captan [N-trichloromethylthio*cyclo*hex-4-ene-1:2-dicarboxyimide]. J. Roubert (Lab. de. Chim.-Œnologie de l'École Nat. d'Agric., Algiers). *Chim. Anal.*, 1959, **41** (8), 333–334.—The method of Kittleson (*cf.* *Brit. Abstr. C*, 1952, 569) has been modified to increase the sensitivity. *Procedure*— Into a test-tube place 1 ml of a $CHCl_3$ soln. of the sample containing up to 50 μg of captan. Add resorcinol soln. (2% in ethanol) (1 ml), place a piece of filter-paper (11 cm × 1 cm) in the soln. and evaporate to dryness under an infra-red lamp. The presence of captan is indicated by a wide band of yellow fluorescence when the paper is viewed by u.v. light. By carrying standards through the procedure it is possible to determine 10 to 50 μg of captan with a precision of 5 μg.
W. T. CARTER

2505. Quantitative determination of parathion in commercial preparations and its detection in internal organs. L. Stoicheva and P. Abrasheva (Higher Med. Inst., Sofia). *Nauch. Trud.*, *VMI, Sofia*, 1958, **5** (2), 121–132.—The method of Hetel and Hellingman for the quant. determination of total and free *p*-nitrophenol (**I**) in commercial preparations of parathion has been adapted for work with a Pulfrich photometer as well as for low concn. of **I**. A standard curve is prepared by means of pure **I**. *Determination of total* **I**— Parathion (0·03 to 0·05 g) is dissolved in 95% ethanol and diluted to 100 ml; N ethanolic NaOH (0·4 ml) is added to 0·3 to 1 ml of this soln. in a test-tube, the mixture is saponified for ≃10 min. in a boiling-water bath, and the liquid is quant. transferred to a calibrated test-tube with a glass stopper, in which it is first diluted with 95% ethanol to 2 ml and then with water to 4 ml. The extinction of the soln. is measured in a 0·5-cm cell with a S43 filter, and the amount of total **I** is read on the standard curve. *Determination of free* **I**—The preparation (≃0·5 g) is dissolved in diethyl ether

(25 ml) and is extracted with 10% aq. Na_2CO_3 soln. (2 or 3×10 ml); the aq. extracts are combined, N NaOH (10 ml) and water are added to 50 ml and then 95% ethanol to 100 ml. The colour is measured in a 0·5-cm cell with a S43 filter. The amount of free I is derived from a standard curve. From the difference between the total and free I the ester-bound I is calculated. Results are given for 8 samples of parathion. Preliminary experiments to extract parathion from internal organs were made and the method of extraction is described. A suitable method for purification of the extract which would allow the direct determination of diethyl p-nitrophenyl thiophosphate to be made has been suggested. A rapid test for demonstrating the presence of parathion in stomach contents is described.   ABSTR. BULG. SCI. LIT.

2506. Determination of residues of O-2:4-dichlorophenyl OO-diethyl phosphorothioate (V-C 13 Nemacide) by cholinesterase inhibition. G. R. Boyd (Res. Dept., Virginia-Carolina Chemical Corp., Richmond, Va., U.S.A.). *J. Agric. Food Chem.*, 1959, **7** (9), 615–617.—V-C 13 Nemacide, which is effective in controlling nematodes, also chinch bugs and other soil-borne insects, and is less phytotoxic than other nematocides, is determined by a modification of the cholinesterase-inhibition method of Giang and Hall (*cf. Brit. Abstr. C*, 1952, 225), after oxidation with HNO_3 to produce the oxygen analogue. Analyses of 16 vegetables and fruits showed the highest residues in root crops, smaller residues in leaf vegetables and beans, and insignificant amounts in fruits.   M. D. ANDERSON

2507. Determination of residues of zinc ethylenebisdithiocarbamate [zineb]. M. C. Kerssen and P. Riepma (Inst. voor Plantenziekentenkundig Onderzoek, Wageningen, Netherlands). *Tijdschr. PlZiekt.*, 1959, **65**, 27–32 (in English).—A simple method for the determination of surface residues of zineb (I) on crops is described, depending on the measurement of the extinction at 434 mμ of Cu diethyldithiocarbamate in the presence of alkali. Interference by Fe^{3+} is suppressed by the addition of $Na_4P_2O_7$. Residues are washed from the sample with water containing a little Teepol, and the additions of reagents are such that the test soln. contains 30 mg of $Na_4P_2O_7.10H_2O$, 2 ml of 0·5 N NaOH and 375 μg of Cu in a final volume of 16 ml. Beer's law is obeyed for amounts of I up to 500 μg.   E. C. APLING

2508. Method for the detection and estimation of Citicide. K. Krishnamurthy, K. S. Srinivasan and S. K. Majumder (Central Food Technol. Res. Inst., Mysore). *J. Sci. Ind. Res., India, B*, 1959, **18** (8), 333–335.—The method described is based on the colour reaction of Citicide (an insecticidal product obtained by chlorinating turpentine) with pyridine and aq. KOH soln. The specific orange colour developed differentiates Citicide from toxaphene and chlordane. Concentrations as low as 30 μg can be determined.   I. JONES

5.—GENERAL TECHNIQUE AND APPARATUS

General

2509. Weight-in-air basis of adjustment of precision weights. P. H. Bigg (Nat. Phys. Lab., Teddington, Middx., England). *J. Sci. Instrum.*, 1959, **36** (8), 359–361.—The advantages and limitations of the system of adjusting and testing weights, on the basis of weighings in air without buoyancy correction, are discussed.   G. SKIRROW

2510. Convenient specimen carrier for use with the quartz-fibre "fishpole" micro-balance. R. S. Thomas (Physiol. Dept., Carlsberg Lab., Copenhagen). *Mikrochim. Acta*, 1959, (6), 831–834 (in English).—The usefulness of the Lowry microbalance (*cf. J. Histochem. Cytochem.*, 1953, **1**, 420), which has a max. load of $\simeq 30$ μg, may be considerably extended by the provision of specimen carriers consisting of small sponges of polystyrene foam for weighing dispersed material. The sponges are weighed, soaked with the suspension or soln. in ethanol or water, dried, and weighed again to give the weight of non-volatile material trapped within the sponges. The polystyrene may be removed by ashing, or separated from the sample by the use of suitable solvents.   D. F. PHILLIPS

2511. Polyethylene micro-pipettes (0·4 to 10 microlitres). H. Mattenheimer and K. Borner (Physiolog.-chem. Inst., Freien Univ., Berlin). *Mikrochim. Acta*, 1959, (6), 916–921 (in German).—Details are given for the rapid and easy construction of these pipettes. Calibration to a definite volume is easier than for constriction pipettes of similar capacity. A relatively low standard deviation ($\pm 0.4\%$) is attainable.   D. F. PHILLIPS

2512. Soil-crushing machine. W. T. H. Williamson (Dept. Soil Sci., Univ., Aberdeen). *Chem. & Ind.*, 1960, (9), 224–225.—A machine is illustrated which is designed for crushing soil samples for analysis without undue breakdown of individual particles and entirely without the production of dust clouds. It is essentially a plate mill with rubber bonded on to the bottom of the bowl and on to the roller.   N. E.

2513. Device for measuring the moisture content of tobacco or like fibrous material. K. Körber [Inventor: O. Peters]. Brit. Pat. 827,708; date appl. 22.7.58. Addn. to Brit. Pat. 827,004, dated 11.1.57.—The duct through which the material is caused to flow, and which includes a conductor surface connected to an external electric circuit to provide a change of capacity dependent on the moisture content, is arranged in a horizontal position and adapted to function as a vibratory conveyor.   J. M. JACOBS

2514. [Continuous] gas-sampling apparatus. W. R. Hamilton. Brit. Pat. 828,151; date appl. 12.11.54.—The apparatus comprises a gas-inlet tube connected to the upper end of a sampling tube, a levelling reservoir connected through flexible tubing to the sampling tube, and mechanical means for lowering the levelling reservoir at a constant rate.   J. M. JACOBS

2515. Apparatus responsive to the composition of a gaseous medium. La Détection Électronique Française (D.E.F.). Brit. Pat. 828,121; date appl.

21.1.58. France, date appl. 24.10.57.—Apparatus for detecting and/or controlling variations in the composition of a gas comprises a detector unit consisting of a first ionisation chamber through which the gas is passed, a second, reference, ionisation chamber connected electrically in series with the first, so as to deliver, at a common junction, a potential depending on the internal resistance of the first chamber. An output unit (comprising a multi-electrode valve), connected to the junction, is responsive to the potential at the junction and delivers a signal to represent the variations in composition of the ionised gas.   The first chamber comprises an electrically conductive outer cylinder maintained at a fixed reference potential, an insulating inner cylinder supporting an ion-collector cathode, a radioactive source, a screen grid disposed beyond the anode in the direction of gas flow, and means for delivering a stream of gas to be tested through the first chamber.   J. M. JACOBS

2516. Absorption of carbon dioxide by soda asbestos and soda lime. G. Kainz and L. Hainberger (II Chem. Inst., Univ., Vienna). *Mikrochim. Acta*, 1959, (6), 870–874 (in German).—A certain amount of water must be present on the surface of soda asbestos before efficient absorption of CO_2 can take place. It is recommended that, before the first run of the day, and also after charging new tubes, 1 to 2 cm of the alkaline asbestos should be warmed slightly in order to draw water from the interior to the surface. The subsequent analyses of a series will not require this pre-treatment since sufficient water is set free when the CO_2 is absorbed.
D. F. PHILLIPS

2517. Apparatus for quantitative gas-absorption measurements. J. R. Shelton and E. T. McDonel. *J. Appl. Polymer Sci.*, 1959, **1** (3), 336–338.—A commercially available aluminium-block heating unit, adapted to accommodate glass sample-tubes, is mounted in an octagonal frame. Gas burettes and levelling bulbs are supported on the frame to provide an apparatus for measuring changes in vol. at a given pressure. Data on the rate of increase of temp. and the temp. distribution within a sample tube are given. The apparatus has been used primarily for volumetric absorption measurements of O in studies related to the oxidation of rubber, but it is adaptable to studies involving other materials and other gaseous atmospheres at controlled temp. *e.g.*, studies have been made with polyethylene, epoxide coatings and drying oils. The apparatus has also proved useful for curing polyurethanes under moisture-free conditions and for the vulcanisation of rubber in an atmosphere of N.
REV. CURR. LIT. PAINT COL. VARN.

2518. Improved micro steam-distillation apparatus. L. R. Fina and H. J. Sincher (Dept. of Bact., Kansas State Univ., Manhattan, U.S.A.). *Chemist Analyst*, 1959, **48** (3), 83.—An all-glass apparatus suitable for the determination of ammonia (Kjeldahl) or of volatile organic acids is described and illustrated.
G. S. ROBERTS

2519. All-glass self-cleansing steam-distillation apparatus. G. S. Duboff (Dept. of Obstetrics and Gynaecology, Univ. of Michigan. Ann Arbor, U.S.A.). *Analyst*, 1959, **84**, 619–620.—The apparatus described and illustrated is a non-jointed single unit consisting of a steam-generating flask, a steam trap, a water-cooled condenser and a vacuum-jacketed reaction vessel. By manipulation of stopcocks the steam-distillate is directed into the condenser and, when distillation is complete, the residual reaction mixture can be sucked into and discharged into the steam trap, and replaced by condensed water from the generator which in turn is discharged through the steam trap, this washing process being repeated as often as is necessary. The apparatus is suited for the determination of a wide variety of substances, *e.g.*, formaldehydogenic corticosteroids, amines distilled from catecholamines, and proteins by distillation of NH_3.
A. O. JONES

2520. Apparatus for suction filtration under inert atmosphere. F. L. Johnson, jun., and G. H. Ayres (Dept. of Chem., Univ. of Texas, Austin, U.S.A.). *Chemist Analyst*, 1959, **48** (3), 82.—A rather intricate glass attachment to a sintered glass crucible is described and illustrated. With care in manipulation the apparatus can be used for quant. work.
G. S. ROBERTS

Chromatography, ion exchange, electrophoresis

2521. Machine for the automatic chromatography and assay of mixtures of radioactive substances. M. C. Corfield, S. Dilworth, J. C. Fletcher and R. Gibson (Wool Ind. Res. Ass., Torridon, Headingly, Leeds, England). *Int. J. Appl. Radiation and Isotopes*, 1959, **5** (1), 42–50.—Constructional details of the machine are described and illustrated. It is arranged that the eluate from a chromatographic column is automatically divided into fractions which are then dried and β-counted; a record of the counts is provided. The machine has been used in the analysis of mixtures of amino acids labelled with ^{131}I and ^{64}Cu, and the methods used are described and discussed.   G. J. HUNTER

2522. A double-fronting phenomenon in one-step development chromatography on anion exchangers. W. Björk (Inst. of Biochem., Univ. of Uppsala, Sweden). *J. Chromatography*, 1959, **2** (5), 536–543 (in English).—The homogeneous compound ε-dinitrophenyl-lysine has been split into two zones by one-step development on diethylaminoethylcellulose with 0·075 N tri(hydroxymethyl)methylamine - HCl buffer of pH 8·9. The phenomenon is due to interaction with atmospheric CO_2 and can be avoided by working with a carbonate-free system; possible mechanisms are discussed. Double-fronting may also appear in gradient development and in chromatography with mixed buffers.
A. R. ROGERS

2523. Colorimetric analysis and recording apparatus and method. Technicon Chromatograph Corp. Brit. Pat. 823,359; date appl. 26.8.57. U.S.A., date appl. 9.10.56.—Measured quantities of liquid from a chromatographic column are collected separately in a receptacle, from which they are delivered successively to a colorimeter for analysis of the liquids in the order in which they were collected in the receptacle.   J. M. JACOBS

2524. A simple arrangement, using a standard Geiger - Müller tube, for the continuous monitoring of radioactive effluents from a chromatography column. H. E. Dobbs (Isotope Research Group, A.E.R.E., Harwell, Berks., England). *J. Chromatography*, 1959, **2** (5), 572–574.—The eluate passes through a brass chamber, one wall of which is the

mica end-window of a Geiger - Müller tube. A polyethylene gasket forms a liquid-tight seal between mica and brass. The capacity of the chamber, including inlet and outlet tubes, is 1·8 ml. The device will satisfactorily detect low-energy β-emitters (such as ^{14}C) at levels as low as 0·0025 μC per ml of organic solvent.   A. R. Rogers

2525. Transistorised drop-counter for chromatographic use. G. Geerinck and J. Hoste (Lab. for Anal. Chem., Univ., Ghent, Belgium). *Talanta*, 1959, **2** (4), 319–321.—The instrument described includes a highly sensitive miniature photo-diode and is adaptable to long or short columns and/or compounds with high or low distribution coeff.
          W. J. Baker

2526. Quantitative evaluation of paper chromatograms and electropherograms without elution. I. Contribution to the technique. F. Franěk and J. Mastner (Res. Inst. of Food Technol., Prague). *Coll. Czech. Chem. Commun.*, 1959, **24** (8), 2646–2655 (in German).—The intensity of the light dispersed by transmission through uncoloured chromatographic paper and paper stained with amaranth (C.I. Food Red 9) has been measured at different angles. The nature of the dispersion in dry untreated paper differs fundamentally from that in paper rendered transparent with benzyl alcohol. From the measurements, the extinctions of the dye in different directions and at different angles are determined, and a mathematical treatment is given.   J. Zýka

2527. Paper-chromatographic analysis of acids. Horizontal migration method. V. V. K. Mohan Rao (Central Drug Res. Inst., Lucknow, India). *Z. anal. Chem.*, 1959, **169** (3), 170–177 (in English).—The relationship between R_F values obtained on straight and circular chromatograms of a few amino and organic acids was investigated. Differences were smaller with collidine as solvent than with n-butanol - acetic acid - water (4 : 1 : 5). The R_F values in 5 basic and neutral solvents were determined on circular chromatograms for about 20 amino acids, and of these the pyridine - water (3 : 1) system gave the best resolution. Better separations were also obtained for circular chromatograms at higher temp.   G. P. Cook

2528. Studies on inorganic paper chromatography. VI. The separation mechanism of cations with butanol - hydrochloric acid as developer. Syoiti Yamada (Chem. Dept., Mie Univ., Tsu). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (5), 485–487. —In the ascending paper-chromatographic separation of Cd, Bi and Cu with a mixture of butanol and 6 N HCl, the R_S value (the ratio of the distances travelled by the front line of the cation and the front line of the solvent) is independent of the concn. of the salts and is in good agreement with the R_F value measured at the front of a spot. It would seem that the migration of cations is mainly governed by partition.   K. Saito

2529. Chromatographic chamber with simple thermal shield. K. Egger and W. Ensslin (Inst. f. angew. Botanik, Univ. Tübingen). *Z. anal. Chem.*, 1959, **169** (1), 16–18 (in German).—A simple chromatographic chamber is described which consists of a cylindrical glass vessel with a metal shield. The filter-paper is inserted in the form of a spiral, and several chromatograms may be developed simultaneously. Diagrams are given.
          S. M. Marsh

2530. New automatic recording densitometer for paper chromatography. B. M. Johnstone and G. P. Briner (Dept. of Pharmacol., Univ. of Melbourne, Australia). *J. Chromatography*, 1959, **2** (5), 513–518 (in English).—The paper chromatogram is illuminated through a slit by light from a 15-watt fluorescent tube and the transmitted light is detected with a 90 AV photo-tube with S4 spectral response. The amplified signal is recorded on a chart stapled to the chromatogram; this ensures a positive 1 : 1 correspondence between record and paper. The sensitivity is high and it is not necessary to make the paper translucent. The over-all accuracy is better than $\pm 3\%$.   A. R. Rogers

2531. Method by which radioactive material may be transferred from a paper chromatogram to a planchette. R. Svendsen (Div. of Endocrinology, Univ. Inst. for Expt. Med., Copenhagen, Denmark). *Int. J. Appl. Radiation and Isotopes*, 1959, **5** (2), 146–147.—The paper is placed in a small funnel through which eluting soln. runs at the rate of 1 ml per hour. The eluate drops on to a heated aluminium planchette with concentric grooves (depth $< 0·01$ mm); a thin film of silicone grease ensures that the soln. dries on the grooved area. Recoveries of [^{131}I]oestradiol p-iodobenzenesulphonate and [^{35}S]p-iodobenzenesulphonylglycine in three experiments with each were 95 and 97%, respectively.   G. J. Hunter

2532. Gas chromatography and its analytical applications. A review. B. A. Rose (D.S.I.R., Lab. of the Gov. Chemist, Clement's Inn Passage, Strand, London). *Analyst*, 1959, **84**, 574–595.— The theory and basic principles of gas chromatography are described, with details of suitable columns, sample-injection systems and detectors. Applications to qual. and quant. analysis are described with brief mention of some special techniques that have been applied to particular problems. (93 references.)   A. O. Jones

2533. Gas - liquid chromatography. P. G. W. Scott (W. G. Pye and Co. Ltd., Cambridge, England). *Chem. Age*, 1959, **82**, 167–169.—Developments in instrumentation are discussed, including the use of the argon ionisation detector.   N. E.

2534. Volatile liquid partition chromatography. R. S. Porter and J. F. Johnson (Californian Res. Corp., Richmond, U.S.A.). *Nature*, 1959, **184** (Suppl. 13), 978–979.—In the method described, which is based on the use of a circular gas chromatograph (*cf. Ibid.*, 1959, **183**, 391), the gaseous phase of the partitioning liquid is used as the eluting gas. This method of using relatively high mol. wt. carrier gases may be of value in non-analytical applications of gas chromatography.
          K. A. Proctor

2535. Characterisation of organic compounds by gas chromatography. II. A precision gas chromatograph. P. Tóth, E. Kugler and E Kováts (Eidg. Tech. Hochsch., Zürich, Switzerland). *Helv. Chim. Acta*, 1959, **42** (7), 2519–2530 (in German).—The requirements of a gas chromatograph capable of giving results reproducible to $\pm 1\%$ are discussed, and such an apparatus is described and illustrated. It is claimed that the minimum detectable amount of decane in the carrier gas (He) is 68 p.p.m. with a commercially available katharometer as detector. The theory of gas chromatography is discussed, and an equation is proposed for determining the

sensitivity of a detector. It is stated that the temp. of the column should be controlled to $\pm 0.1°$, that of the flow-meter to $\pm 0.4°$, and the carrier gas pressure should be constant to $\pm 0.5\%$.

III. Calculation of retention indices for aliphatic, alicyclic and aromatic compounds. A. Wehrli and E. Kováts. *Ibid.*, 1959, **42** (7), 2709–2736 (in German).—The "retention index" (I) is defined, and it is shown that $\triangle I$, *i.e.*, the difference between I for a polar stationary phase and I for a non-polar one, is often an additive structural property. Examples are given of the elucidation of correct structures of compounds when alternatives exist. A comprehensive list of retention indices is given for over 200 organic compounds at 70°, 130° and 190°, both for the polar phase Emulphor-O (a polyoxyethylene glycol fully etherified with octa-decanol) and for the non-polar phase Apiezon L. These indicate that I for paraffins is independent of the stationary phase.   H. M.

2536. Design considerations of a gas-chromatography system employing high-efficiency Golay columns. R. D. Condon (The Perkin-Elmer Corp., Norwalk, Conn., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1717–1722.—The use of Golay columns and ionisation detectors for separating samples either with a wide range of boiling-points, or within a narrow range, is described. Samples with low vapour pressures can also be analysed.
                                    K. A. PROCTOR

2537. The potentialities of coated capillary columns for gas chromatography in the petroleum industry. D. H. Desty, A. Goldup and B. H. F. Whyman (B.P. Co. Ltd., Sunbury, Middx., England). *J. Inst. Petrol.*, 1959, **45**, 287–298.—Apparatus, operable up to 250°, incorporating a flame ionisation detector and a novel sample-introduction system for both gaseous and liquid samples, is described. The performances of metal and glass columns are tabulated. Stress is laid on the importance of small amounts of material and short operating time. An efficiency of 117,000 plates was recorded with a copper capillary (0.028 cm internal diam., 82 metres long, wound into a coil on a metal spool 2 in. in diam. and 3 in. long). By using a coating of squalane and with nitrogen (0.6 ml per min.) as the carrier gas at 72°, a mixture (2 μg) containing 17 of the 18 possible isomeric C_8 paraffins was resolved into 14 maxima in a total analysis time of 7 min. The theory of coated capillary columns is discussed and the Golay and van Deemter equations for the height equivalent to a theoretical plate are compared. The absence of "inert support" in coated columns does not give the "no tailing" advantage expected.   E. G. CUMMINS

2538. Influence of pressure gradients on resolution in gas chromatography. G. H. Stewart, S. L. Seager and J. C. Giddings (Dept. of Chem., Univ. of Utah, Salt Lake City, U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1738.—A modified equation is evolved for the H.E.T.P. of a gas-chromatographic column and is useful in giving a theoretical basis for pressure gradient effects.   K. A. PROCTOR

2539. Supports for gas - liquid separation chromatography. M. Kubínová (Res. Inst. Synth. Rubber, Gottwaldov, Czechoslovakia). *Chem. Listy*, 1959, **53** (8), 850–856.—When testing 14 types of material used as inert supports it was found that these do not differ in efficiency for the determination of non-polar compounds (hydrocarbons). For the determination of polar compounds (alcohols), supports with a large pore size are recommended.
                                    J. ZÝKA

2540. Modification of "Agla" micrometer hypodermic syringe for use in vapour-phase chromatography. J. W. Sweeting (Commonwealth Sci. and Ind. Res. Organisation, N.S.W., Australia). *Chem. & Ind.*, 1959, (37), 1150.—To avoid the spreading of the peaks that results from protracted addition times, a modified micrometer barrel has been designed for the instantaneous addition of larger samples (>0.01 ml). The quantity to be added is pre-set with the micrometer scale and is then rapidly injected by pushing a plunger to the end of its travel.   E. G. CUMMINS

2541. Determination of surface area using a gas chromatograph. J. F. Roth and R. J. Ellwood (Central Res. Lab., General Aniline & Film Corp., Easton, Pa., U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1738–1739.—A number of modifications to the method of Nelsen and Eggertsen (*Anal. Chem.*, 1958, **30**, 1387) that simplify the operating procedure and calculations are described.
                                    K. A. PROCTOR

2542. Carrier electrophoresis with higher potentials. K. Dose (Max Planck Inst. für Biophysik, Frankfurt am Main, Germany). *J. Chromatography*, 1959, **2** (5), 575–577 (in German).—The process of electrophoresis on blocks of paper pulp or starch requires special provision for the dissipation of heat when high potential gradients are used. In the apparatus described, the medium is supported on a liquid-cooled plate of copper or brass, which is covered with a thin film of a plastic material with appropriate electrical and chemical properties.
                                    A. R. ROGERS

See also Abstracts—**2318, 2320, 2321,** Gas chromatography of hydrocarbons.

Optical

2543. Use of scintillation counters in single crystal X-ray diffractometry. P. J. Black and J. B. Forsyth (Crystallographic Lab., Cavendish Lab., Cambridge, England). *J. Sci. Instrum.*, 1959, **36** (9), 392–394.—An examination of the signal-to-noise ratio as a function of the operating potential of a photo-multiplier (DuMont 6291 tube) shows that the resolution of signal pulses from background noise improves as the potential is decreased. Signal resolutions of 26, 29 and 43% have been obtained with AgK_α, MoK_α and CuK_α radiations, respectively, the mean number of photo-electrons released per keV of incident quantum energy being 6.
                                    G. SKIRROW

2544. Use of X-ray diffraction analysis in the study of equilibrium systems. F. Kupka (Inst. Crude Minerals, Kutná Hora, Czechoslovakia). *Chem. Listy*, 1959, **53** (8), 793–804.—The principles and a survey of the method are given, with 98 references. Special attention is paid to the preparation of the samples and to the use of high-temp. X-ray diffraction analysis.   J. ZÝKA

2545. "Vacuum cup" electrode for the spectrochemical analysis of solutions. T. H. Zink (Illinois Inst. of Technology, Chicago, U.S.A.). *Appl.*

Spectroscopy, 1959, **13** (4), 94–97.—In the electrode described, which can be made from pure copper, silver, graphite or other materials, the sample is excited in the form of a very fine spray. The rate of transfer of the soln. into the electrode gap can be varied from <1 ml per 100 sec. to 1 ml per 40 sec. and the vol. of sample used may range from 0·2 to 4 ml. The method is speedy and sensitive and requires shorter exposure times than do other solution techniques.   K. A. PROCTOR

2546. Low-voltage pulse discharge *in vacuo* to obtain spectra. F. Z. Pedos, N. S. Sventitskiĭ and Z. I. Shlepkova. *Optics and Spectroscopy*, 1959, **6** (6), 533–534; English translation of *Optika i Spektroskopiya*, 1959, **6** (6), 815.—A circuit is described which enables a low-voltage discharge to operate at very low pressure (10^{-4} to 10^{-5} torr) between solid electrodes separated by a gap of several mm. The radiation emitted from this discharge can serve as a source for a number of spectroscopic determinations.   B. S. COOPER

2547. Construction of variable-slit photometers for the measurement of effective widths. E. W. Salpeter (Lab. Astrofisico, Specola Vaticana, Castel Gandolfo). *Metallurg. Ital.*, 1958, **50** (8), 293–295.—The construction of a simple precision variable slit, which opens to a width of 10 to 15 mm., is described. It consists of a hinged parallelogram, to two of the longer sides of which are fixed the plates that form the slit. A photometer for measuring effective line widths can be constructed by combining the variable slit with a densitometer. A complete variable-slit photometer is obtained by combination with a densitometer and any spectrum-projection system.   J. H. WATON

2548. Influence of the photographic emulsion on effective width. A. Lais and R. Intonti (Lab. di Chim., Ist. Sup. di Sanità, Roma). *Metallurg. Ital.*, 1958, **50** (8), 296–300.—When the curves of log of effective line width *vs.* log of exposure time are plotted, emulsions of fine grain give the greater slope.   J. H. WATON

2549. Possibilities of employing effective widths. A. Fiorenza and M. Lachin (Lab. Gomma, Pirelli S.p.A.). *Metallurg. Ital.*, 1958, **50** (8), 319–324.—The use of effective line width (*cf.* Junkes and Salpeter, *Anal. Abstr.*, 1955, **2**, 1741) is studied in the analysis of powdered materials in the high concn. range. The procedure gives an extension of the analytical curves, compensates for variation of emission and is largely independent of the characteristics of the photographic emulsion.

                    J. H. WATON

2550. Effects of the variation of the ambient pressure on the intensity of arc emission of some elements. G. Brautti (Ist. Sper., Talassografico, Trieste). *Metallurg. Ital.*, 1958, **50** (8), 340–342.—The variation in the intensity of emission lines with change in pressure is investigated with electrodes of copper and zinc in atmospheres of N, Ne, A, CO_2 and air.   J. H. WATON

2551. Advantages of selective volatilisation and of "enhancement" in spectrographic analysis. G. Rigault (Ist. Mineralog. e Petrograf., Univ., Torino). *Metallurg. Ital.*, 1958, **50** (8), 301–304.—Among the possible ways of raising the sensitivity of spectrographic analysis with a d.c. arc, that of

altering the conditions of excitation of the luminous source is considered. By using two arcs, one acting as a heat source and the other as a light source, lines due to volatile elements may be excited selectively by a process of "selective distillation"; also, the amount of elements of low volatility on the electrode may be increased by removing the volatile elements from successive additions of the sample. The sensitivity of the analysis can also be improved by controlling the phenomenon of "enhancement." By incorporating an equal weight of Na_2CO_3 with the sample, the presence of an element of low ionisation potential (*e.g.*, Na) will enhance other lines of low excitation energy; the presence of Na_2CO_3 also permits the elimination of lines due to cyanogen. This technique should prove of great value in geochemical studies.   J. H. WATON

2552. Determination of the degree of absorption of analytical lines in arc and spark spectra. I. V. Podmoshenskiĭ and V. M. Shelemina. *Optics and Spectroscopy*, 1959, **6** (6), 531–532; English translation of *Optika i Spektroskopiya*, 1959, **6** (6), 813.—A method is described for measuring self-absorption in the lines used for spectrochemical analysis, the excitation conditions being chosen so as to suit the desired analytical procedure. Data are presented for some commonly used lines of Ni, Cr, Fe and Si.

                    B. S. COOPER

2553. Spectrometer sample heater. T. E. Lusk (Light Military Electronics Dept., Adv. Electronics Center of the General Electric Co., Ithaca, N. York). *Rev. Sci. Instrum.*, 1959, **30** (8), 748–749.—The heater is in two halves. Each consists of 12 in. of molybdenum wire (0·3 in. in diam.) passed through ceramic insulators which are sandwiched between two copper plates. The sample is clamped between the two halves and temp. of 400° can be attained with an input of about 5 V.   G. SKIRROW

2554. Differential micro-densitometer. A. H. Cook and R. G. Hitchins (D.S.I.R., Nat. Phys. Lab., Teddington, Middx., England). *J. Sci. Instrum.*, 1959, **36** (8), 337–341.—The principle of a micro-densitometer, by means of which instrument both density and rate of change of density of photographic plates can be measured, is discussed and details are given of the instrument design.   G. SKIRROW

2555. Spectrum display comparator. W. J. Price (Magnesium Elektron Ltd., Manchester). *Brit. J. Appl. Phys.*, 1959, **10** (8), 352–355.—The instrument described permits the spectra of the sample and standard spectra to be projected in juxtaposition on a screen, simultaneously with the production of an oscillographic trace of the spectrum-density profile. It is particularly useful for determining trace elements under adverse spectral conditions.

                    S.C.I. ABSTR.

2556. Highly sensitive recording micro-spectrophotometer. B. Chance, R. Perry, L. Åkerman and B. Thorell (Johnson Res. Found., Univ. of Pennsylvania, Philadelphia). *Rev. Sci. Instrum.*, 1959, **30** (8), 735–741.—Details are given of an instrument capable of measuring the light absorption of substances present at "enzyme" concn. in a single living cell. The circuitry is adapted for measurement of small photo-currents at high signal-to-noise ratios. Its application to the study of the mitochondrial aggregate of the grasshopper spermatid is described.

                    G. SKIRROW

2557. Interference filters for the ultra-violet region of the spectrum. R. S. Sokolova and T. N. Krylova. *Optics and Spectroscopy*, 1959, **6** (6), 513–515; English translation of *Optika i Spektroskopiya*, 1959, **6** (6), 788.—Multilayer reflection filters suitable for use in the u.v. region are made by depositing alternate layers of ThO_2 and SiO_2 on the surface of a plate of fused SiO_2. Peak reflection factors may be as high as 95%. Combinations of these filters with conventional ones may be used to obtain relatively high spectral transmissions over restricted spectral ranges. A modification of the layer structure, using the same two oxides, yields interference filters for the 250 to 400-mμ range having peak transmissions of up to 80% and half-widths of 6 to 14 mμ. These filters are sufficiently stable not to require protection from the atmosphere.
B. S. COOPER

2558. Grating spectrophotometer for the Schumann ultra-violet range. F. J. P. Clarke and W. R. S. Garton (A.E.R.E., Harwell, Berks., England). *J. Sci. Instrum.*, 1959, **36** (9), 403–409.—Details are given of an instrument for the measurement of the optical transmittances of small solid specimens in the range above 900 A. The concave grating is of 1 metre radius and 600 lines per mm, and scanning of the spectrum across the slit is carried out by linear movement of the centre of the Rowland circle.
G. SKIRROW

2559. Solvents for ultra-violet spectrophotometry. D. D. Tunnicliff (Shell Development Co., Emeryville, Calif., U.S.A.). *Talanta*, 1959, **2** (4), 341–347. —The effect of absorption of light by the solvent on the accuracy of u.v. absorption measurements is examined theoretically, and the source and purification of the usual solvents are discussed. No exact specification for solvent transmittance can be established. A procedure is given for purifying *iso*octane by treatment with N_2O_4, followed by percolation through a column of Ascarite and silica gel. Traces (<0·1%) of highly branched olefins can thus be removed, and the pure solvent is then satisfactory down to $\simeq 2100$ A (1-cm cell) or 1900 A (0·1-cm cell). A method of purifying dioxan by distillation from alkaline $KMnO_4$ is also described.
W. J. BAKER

2560. Hydrogen and mercury - helium light sources for SF-4 spectrophotometers. S. I. Levikov and L. P. Shishatskaya. *Optics and Spectroscopy*, 1959, **6** (5), 446–448; English translation of *Optika i Spektroskopiya*, 1959, **6** (5), 688.—The spectral range of the Russian SF-4 quartz photo-electric spectrophotometer is 220 to 1100 mμ. The two standard sources to cover this range are a tungsten-filament lamp and a hydrogen discharge lamp. In addition, a mercury - helium discharge lamp is supplied and used for wavelength calibration. The constructional details, operating circuits and radiation characteristics of the two discharge lamps are described.
B. S. COOPER

2561. New absolute wedge-calibration method for the Dobson ozone spectrophotometer. J. P. Funk (C.S.I.R.O., Div. of Meteorolog. Phys., Aspendale, Australia). *J. Sci. Instrum.*, 1959, **36** (8), 378.—A perforated plate, having 148 holes of diam. 0·076 in., is mounted on top of the tubular fitting of the standard lamp-holder. Above this is a second plate having 74 slightly larger holes positioned so as to obscure every second hole in the bottom plate. When calibrating, the top plate is removed and a

reading is taken through the full number of holes; the top plate is then replaced, and two readings are taken through alternate sets of holes. Finally, another reading is taken through the full number of holes.
G. SKIRROW

2562. Microscope - spectrophotometers. L. S. Agroskin and N. V. Korolev. *Optics and Spectroscopy*, 1959, **6** (6), 544–545; English translation of *Optika i Spektroskopiya*, 1959, **6** (6), 832.—Two arrangements are described in which a microscope and spectrophotometer are combined for the determination of the absorption, reflection or fluorescence spectrum of a very small sample. These have been applied successfully to the determination of the visible and u.v. spectral characteristics of biological specimens (including single cells), microcrystals, and spots on paper chromatograms.
B. S. COOPER

2563. Low-temperature infra-red cell. C. M. Lovell and H. F. White (Res. Dept., Union Carbide Chemicals Co., S. Charleston, W. Va., U.S.A.). *Appl. Spectroscopy*, 1959, **13** (4), 108–109.—The cell described can be adapted from any commercial i.r. liquid cell and is particularly suitable for the study of materials which are unstable at room temp. It can also be used to obtain the spectra of low-boiling liquids without resorting to expensive pressure cells.
K. A. PROCTOR

2564. Potassium bromide disc holder for Beckman DU spectrophotometer. W. H. Waggoner (Dept. of Chem., Univ. of Georgia, Athens, U.S.A.). *Chemist Analyst*, 1959, **48** (3), 80–81.—The construction and operation of the holder are described. It can be made from brass, aluminium or stainless steel, and accommodates four 0·5-in. discs.
G. S. ROBERTS

2565. Effect of absorbancy on Raman intensities in solutions. F. Vratny and R. B. Fischer (Dept. of Chem., Univ. of Indiana, Bloomington, U.S.A.). *Talanta*, 1959, **2** (4), 315–318.—A study of data obtained on aq. soln. of H_2SO_4 and H_3PO_4 shows that the observed intensities of the Raman lines agree reasonably well with those calculated from an equation based on the laws of absorbancy. The Raman intensity varies directly with concn. when there is excitation outside an absorption band, but inversely (over some or all ranges of concn.) when the excitation is within the band. For simple quant. analysis by Raman spectroscopy, spectral regions wherein the sample shows appreciable absorption should be avoided.
W. J. BAKER

2566. X-ray fluorescence spectroscopy. Applications of a simple apparatus using radioactive sources. C. E. Mellish (Isotope Res. Div., A.E.R.E., Harwell, Berks.). *Research*, 1959, **12** (6), 212–217.—The X-ray tube is replaced by a radioactive source and the crystal spectrometer by a proportional or scintillation counter. Suitable sources are ^{241}Am, ^{55}Fe, ^{3}H or ^{181}W. The apparatus is well suited to routine work such as the determination of metal-plating thickness or of Mo in steel.
N. E.

2567. Solution techniques in fluorescence X-ray spectrography. W. J. Campbell, M. Leon and J. W. Thatcher (Bureau of Mines, College Park, Md.). *U.S. Bur. Mines, Rep. Invest.*, 1959, No. 5497, 24 pp.— The best materials for the construction of soln. cells are discussed. The effects of variations in the nature and concn. of the solvent acid are investigated and

it is concluded that dil. soln. in HNO_3 are preferable. Normally only one comparison standard is required, since there is a linear relationship between intensity and concn. in dil. soln.   Matrix effects are effectively eliminated by the dilution technique or by the use of internal standards.   Limits of detection are listed and are generally between 0·02 and 0·002 g per litre.   Three practical applications are described, *viz*, Mn and Fe in a low-grade manganese ore, Fe in a low-grade copper ore, and Ca in wolframite concentrates.   P. T. BEALE

2568. New standardisation technique for X-ray absorption measurements.   J. O. Hibbits, S. S. Cooper and M. R. Menke (Aircraft Nuclear Propulsion Dept., General Electric Co., Cincinnati 15, Ohio, U.S.A.).   *Anal. Chem.*, 1959, **31** (10), 1748.— Aluminium plates about 0·1 in. thick can be standardised against standard uranium soln. and thereafter used instead of these soln., thus saving considerable time and effort in the determination of U by X-ray absorption measurements when frequent calibration is necessitated by changes in instrument response.   K. A. PROCTOR

2569. Comparator for X-ray spectrum films. C. F. Smith (Farnham Common, Bucks., England). *J. Sci. Instrum.*, 1959, **36** (8), 377–378.—A simple instrument is described which enables films or plates of spectra to be quickly compared.

G. SKIRROW

2570. Use of a photomultiplier in the flame-photometric analysis of substances with emission bands in the red.   E. J. Harris (Biophys. Dept., Univ. Coll., London).   *J. Sci. Instrum.*, 1959, **36** (8), 369.—The EMI Electronics photomultiplier E 9578 TA is suitable for measuring the emissions of lithium, potassium and rubidium up to 7800 A. By using a Beckman atomiser burner, a Hilger glass monochromator and this tube, quantitative measurements can be made on soln. containing 0·1 to 0·5 μmole of RbCl (or KCl) per ml.

G. SKIRROW

1571. Improved assembly for filtering solutions for flame-photometric analysis.   L. C. Blakemore and M. Fitzpatrick (Soil Bur., D.S.I.R., Wellington, N. Zealand).   *Chemist Analyst*, 1959, **48** (3), 82–83. —A modification of the technique of filtering under suction through paper supported by sintered glass is described.   A Soxhlet extraction thimble is fitted inside a tall, sintered glass crucible and will function for about 30 filtrations.   G. S. ROBERTS

2572. Photo-electric recording interferometer for gas analysis.   Susumu Namba (Inst. of Phys. and Chem. Res., Komagome, Bunkyo-ku, Tokyo, Japan). *Rev. Sci. Instrum.*, 1959, **30** (8), 642–645.—The central part of the interference fringes is projected through a polariser (P_1) on to the slit of a photomultiplier.   The principal axis of the polariser is perpendicular to the fringes and a field-splitting polariser (P_2), the two planes of which are split by a line parallel to the fringes and oriented at $\pm 45°$ to the principal axis of P, and a rotating Polaroid disc.   The shift of the fringe centre relative to the splitting line of P_2 generates an alternating photo-current which is a function of the shift.   G. SKIRROW

Thermal

2573. The reading of calorimeter thermometers. I. Davies (Central Electricity Generating Board, Battersea, London).   *Chem. & Ind.*, 1959, (37), 1151.—This statistical survey emphasises the need for special precautions when estimating 0·001° on such thermometers as the 6° calorimeter thermometer, where the smallest graduation is 0·01°. The use of a telescope bearing an eyepiece graticule is advocated.   E. G. CUMMINS

2574. Simple recording thermobalance for vacuum and pressure studies.   J. G. Rabatin and C. S. Card (Advance Devel. Lab., General Electric Co., Cleveland, Ohio, U.S.A.).   *Anal. Chem.*, 1959, **31** (10), 1689–1692.—The thermobalance described can be operated at pressures < 1 torr, at atmospheric pressure and up to 40 atm. pressure of CO_2, N, H_2S, CO, O and NH_3.   It consists of a modified torsion balance, with a photocell - light transducer system. The max. sensitivity is about $\pm 0·2$ mg for weight changes of 100 to 200 mg.   The balance is comparable in performance with the Chevenard thermobalance, and is superior for fast reaction systems.

K. A. PROCTOR

2575. Improved vacuum fusion, gas extraction, and collection apparatus.   A. Lench and G. S. Martin (Defence Standards Lab., Maribyrnong, Victoria, Australia).   *Anal. Chem.*, 1959, **31** (10), 1726–1729.—The apparatus described for the determination of gases in metals features a large crucible, a high-speed diffusion pump for rapid gas transport, provision for gas collection and measurement between 0·05 and 38 ml, an improved O-ring vacuum valve, solenoid-operated glass - mercury valves, and a butt-jointing method of assembly for the rapid removal and replacement of defective components.   The apparatus has been used successfully for the determination of the gas content of a wide variety of chromium and titanium metal and alloy samples with a steel bath as the reaction medium.   K. A. PROCTOR

2576. Effect of evaporated films on recovery of gases evolved during vacuum fusion analyses. A. L. Beach and W. G. Guldner (Bell Telephone Lab. Inc., Murray Hill, N.J., U.S.A.).   *Anal. Chem.*, 1959, **31** (10), 1722–1726.—The error introduced through the sorption of gas by metal vapour or films deposited on the furnace walls during a fusion analysis has been evaluated.   The effects of C, Ni, Fe, Ge, Pt, Al, Mn and Ti films on the recovery of CO, H and N have been studied.   These data are useful in interpreting the significance of fusion analyses, especially when the gas content is of the order of a few p.p.m.   K. A. PROCTOR

Electrical

2577. Direct-recording electronic polarograph.   G. Horn (Inst. Mikrobiol. u. Therapie, Jena, Germany). *Chem. Tech., Berlin*, 1959, **11** (8), 448–450.—Current from an electronic polarograph (*cf.* Horn and Sickert, *Ibid.*, 1955, **7**, 717) is directly recorded, after amplifying, by a pen recorder, which is quicker and more convenient than photographic recording. The response is sufficiently rapid to show the pulse of current due to each drop of mercury.   The p.d. at half-range during the discharge of an ion is

measured separately within ± 1 mV by a compensation method (*cf.* Gardner, *Nature*, 1951, **167**, 158) after adjusting the voltage to give the same current as that flowing during the recording.

A. R. PEARSON

2578. Studies in polarographic analysis. XXXII. Chronopotentiometry with a dropping mercury electrode. Masayoshi Ishibashi, Taitiro Fujinaga, Atsuyoshi Saito and Kosuke Izutsu (Chem. Dept., Fac. of Sci., Kyoto Univ., Sakyo-ku, Kyoto). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (5), 478–482.—The use of a slow dropping mercury electrode ($\simeq 50$ sec. per drop) is advantageous for the determination by chronopotentiometry of a very small amount of ions in the presence of a large amount of more easily reducible substance (Ishibashi *et al., Bull. Chem. Soc. Japan*, 1958, **31**, 887). The potential - time diagram was examined both theoretically and experimentally for Cd and Pb, and it was confirmed that the presence of a more easily reducible substance (*e.g.*, Pb) does not affect the wave on the diagram. The determination of 10^{-5} and 10^{-6} M Cd in the presence of, respectively, 10^{-3} and 10^{-4} M Pb is effected in 0·5 M HCl in the presence of polyacrylamide (0·01%) as max. suppressor.

K. SAITO

2579. Use of the dropping mercury electrode to estimate oxygen in dilute gels. E. J. K. Penikett (Guy's Hosp. Med. Sch., London, England). *J. Polarographic Soc.*, 1959, (2), 34–37.—An apparatus and procedure for measuring the O concn. at different depths below the surface of semi-solid nutrient media are described.

H. F. W. KIRKPATRICK

2580. Purification of tetrabutylammonium iodide for polarographic use. L. Silverman and W. G. Bradshaw (Atomics International, North American Aviation Inc., Canoga Park, Calif.). *Anal. Chem.*, 1959, **31** (10), 1672.—Dissolve commercially pure tetrabutylammonium iodide (5 g) in methanol - acetone (1:3) (50 ml), filter through paper and allow the filtrate to evaporate at room temp. to a vol. of $\simeq 25$ ml. Add H_2O (5 ml) and dry the pptd. crystals in a vacuum desiccator. The yield is $\simeq 65\%$.

A. R. ROGERS

2581. Quick and anaerobic pH measurements [of blood] at 37°. W. Heerspink and E. J. van Kampen (Klin.-Chem. Lab. Diakonessenhuis, Groningen, Netherlands). *Chem. Weekbl.*, 1959, **55** (34), 453–454.—The apparatus described has been developed for the determination of the pH of blood. The measurement is carried out in a thermostatically controlled vessel at 37°. A Beckman-G blood electrode (glass - calomel) is used as a cell. This electrode is combined with a stopcock system in such a way that every liquid can be measured anaerobically. One measurement requires about 1 ml of liquid. The precision is within 0·02 pH unit. The procedure is given in detail.

M. J. MAURICE

2582. Glass electrodes for pH measurements at high temperatures. B. Polej and F. Štráfelda (Dept. Phys. Chem. Methods, School of Chem.

Technol., Prague). *Chem. Listy*, 1959, **53** (8), 805–810.—A survey is presented of the practical evaluation of, and of tests with, various types of glass used for the preparation of electrodes.

J. ZÝKA

2583. Precise coulometric titration of acids and bases. J. K. Taylor and S. W. Smith (Gen. Motors Corp., Anderson, Ind., U.S.A.). *J. Res. Nat. Bur. Stand., A*, 1959, **63** (2), 153–159.—Apparatus and procedures for the titration of strong and weak acids and bases by constant-current coulometry are described. The cell consists of a platinum cathode and silver anode, with N KCl (for acids) or N Na_2SO_4 (for bases) as supporting electrolyte. The current is adjusted to 200 mA and the soln. electrolysed until near the end-point; the titration is completed at 10 mA, the end-point being determined by a differential potentiometric plot. Titrations of benzoic acid, K H phthalate, adipic acid, HCl and Na_2SO_3 show standard deviations from 0·003 to 0·010 (5 to 8 analyses on each sample). The method is absolute because the results are based on electrical standards without reference to the purity of chemical primary standards.

W. J. BAKER

2584. Instrument for the continuous measurement of dissolved oxygen in water. M.-O. Macklin (Reymersholms Gamla Ind. A.B. Hälsingborg, Sweden). *TVF*, 1959, **30** (7), 265–279.—Theoretical considerations are discussed and the apparatus is described and illustrated.

N. E.

2585. Radio-assay of aqueous solutions. A. Marcó, J. C. Scott, J. C. Elwood and J. T. Van Bruggen (Dept. of Biochem., Univ. of Oregon Med. Sch., Portland, U.S.A.). *Anal. Chem.*, 1959, **31** (10), 1746–1747.—A liquid-sample counting technique that provides a high degree of accuracy and ease of manipulation, and saves both time and materials, is described. The method of sample preparation is less sensitive than some others and is used only when adequate activity is available. The activity (μC) of a series of samples, counted both as liquid and after combustion to CO_2 and plating as $BaCO_3$, and of a series of samples prepared from a single soln. agreed to within less than the 6% counting error expected.
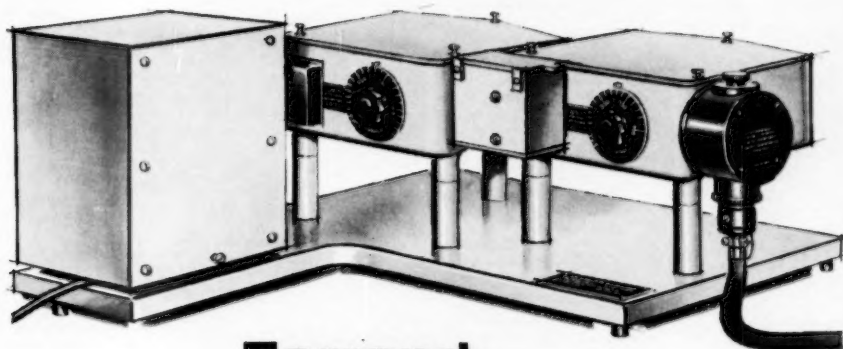
K. A. PROCTOR

2586. Gamma spectrometry for analysis of mixtures of radio-isotopes in biological and medical research. K. J. Öbrink and H. R. Ulfendahl (Inst. of Physiol., Univ of Uppsala, Sweden). *Int. J. Appl. Radiation and Isotopes*, 1959, **5** (2), 99–105. —Both components in mixtures of ^{51}Cr and ^{131}I, and of ^{24}Na and ^{42}K, have been determined even when the count rates were as low as 150 counts per min. A well-type sodium iodide crystal detector was used with a single-channel pulse height analyser; the activities were calculated from the count rates in two channels adjusted with respect to central energy and width to suit the particular measurements; a third measurement on the whole spectrum may be made to enable long-term changes in the apparatus to be eliminated. The stability requirements of the equipment are discussed.

G. J. HUNTER

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list.   When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	A
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
Colour Index	C.I.
concentrated	conc.
concentration	concn.
constant	const.
crystalline	} cryst.
crystallised	
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA

milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecule-e, -ar	mol.
normal (concentration)	N
optical rotation	α_{λ}^{t}
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_{λ}^{t}
relative band speed	R_{F}
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_{\lambda}^{t}$
square centimetre	sq. cm.
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\not>$	not less than	$\not<$
is proportional to	∞	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}.   Substances in the ionic state are represented by Na$^+$, Fe^{2+}, Fe^{3+}, etc., for cations and by Cl$^-$, SO$_4^{2-}$, PO$_4^{3-}$, etc. for anions.

ANALYTICAL ABSTRACTS

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